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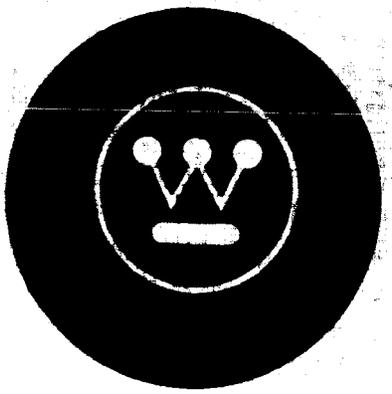
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PROPERTIES OF P-03 GRAPHITE

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PROPERTIES OF P-03 GRAPHITE

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ABSTRACT

P-03 graphite, a premium fine-grained material, has been evaluated for NERVA reactor hardware. This grade is available both as regularly molded stock with a well-established fabrication technology and as semi-commercial isostatically molded stock. Both versions of P-03 have a high and relatively uniform density in the  $1.85 \pm 0.03$  g/cc range. The mechanical properties, shown below for the typical off-the-shelf stock, are very promising for structural applications:

<u>METHOD OF LOADING</u>	<u>NO. OF SPECIMENS</u>	<u>STRENGTH (psi)</u>		
		Low	Average	High
Tension	20	3,700	4,700	5,750
Compression	50	14,400	20,000	22,800

Youngs Modulus is in the  $1.5-2 \times 10^6$  psi range. The thermal expansion coefficient, averaged between room temperature and  $800^\circ\text{C}$  ( $1473^\circ\text{F}$ ), is between 2.3 and 3.3 microinches per inch per  $^\circ\text{F}$ ; for all types of P-03 at higher temperatures the coefficient increases to a somewhat greater value. Electrical resistivity has also been determined; it shows good correlation with flexure strength.

P-03 has a fine grained microstructure; the pores between coke particles are small in comparison with those of most other premium graphites. The large-scale structure is essentially free of such defects as cracks, voids, and inclusions. Application of P-03 graphite for reactor hardware should result in major improvements. This report is intended primarily as a source of necessary data for the design and structural analysis purposes.

## INTRODUCTION

### General Remarks

To meet the structural graphite requirements of the NERVA reactor and maintain a wide margin of safety, a large number of commercial graphites have been surveyed to determine the best product for each hardware application.<sup>1, 2, 3</sup> These surveys disclosed that the highest strength properties were possessed by those graphites made from a small-particle mix formulation; however, such graphites could not be fabricated in large shapes. Therefore, the most promising graphite for a particular application is that manufactured in a relatively small size, yet large enough to produce the hardware component. In addition, the molded graphite grades are preferred over extruded grades due to their better strength and flaw-free structure.

As a result of this survey, one grade of graphite was selected as the most promising material for potential NERVA applications.<sup>4</sup> This was a molded grade P-03, produced by the Pure Carbon Company, Inc.<sup>5</sup> The reasons for selecting P-03 were the following:

1. Excellent strength properties in tension, flexure, and compression.
2. High density and the associated good surface finish after machining.
3. Flaw-free microstructure and low impurity content.
4. Well-established fabrication technology.
5. Manufacturer's capability for the scale-up in product size.
6. Availability of both regularly molded and isostatically molded products.
7. Good radiographic uniformity.

This grade was subsequently recommended for plunger pins, replacing in this application an extruded graphite in which cracks and flow lines were frequently encountered.

This report surveys the physical and mechanical properties of P-03 graphite for the benefit of those dealing with structural graphite hardware in all its phases: design, machining,

quality control, and performance evaluation. It is expected that the use of this high quality graphite will eventually be widened to include other graphite hardware components in the NERVA reactor.

### Description of Material

Grade P-03 is a premium-quality molded graphite. The coke particle mix and the exact nature of the binder are considered proprietary by the manufacturer. No re-impregnation treatment is known to be involved. The average particle size does not exceed 0.003" as compared to 0.006" for ATJ and 0.033" for H4LM molded graphites. With a smaller grain size, the graphite's mechanical properties become more attractive, and hence this grade has been considered for those applications requiring a refractory material with excellent strength.<sup>4</sup>

The manufacturer produces P-03 by both conventional and isostatic molding techniques. This report will emphasize the conventionally molded stock used for the plunger pin hardware. The isostatically molded stock is a potentially excellent structural material for reactor applications when large monolithic pieces are required. This scaled-up isostatic P-03 will be treated separately in a future report; this material has been successfully fabricated in six-foot lengths.

Regularly molded stock is fabricated in both plate and cylindrical geometries (Figure 1a). The regular plate stock size is produced to the maximum dimensions of 12 x 12 x 3 inches. Cylindrical stock is generally 4.8 inches in diameter by 4.8 inches long. The manufacturer provides other shapes (notably as rough-molded hardware configurations) on special order. Owing to the dense, fine-grained microstructure, the thickest cross section that can be processed successfully at this time through the carbonizing and graphitizing bake cycles is about eight inches; in the case of the isostatically molded version, for example, the semi-commercial product is about 7 1/4 inches in diameter and 21 inches long.<sup>4</sup>

Evaluation tests on P-03 graphite have been conducted on the following samples:

A. Isostatically Molded Stock

Sample No. 1: This was received as a machined cylinder 6.8 inches in diameter and 2 inches thick. It had evidently been trimmed from a larger pressing.

Sample No. 2: This sample, roughly one-third of an isostatic pressing, was 7 1/4 inches in diameter and 7 inches long.

B. Regularly Molded Stock

Sample No. 3: This consisted of trimmed plates 2 x 6 x 1 1/4 inches in size, whose original dimensions are uncertain. Untrimmed plates 4 x 4 x 1.1, 6 x 6 x 2, and 12 x 12 x 1.2 inches in size were also included.

Sample No. 4: This sample consisted of cylindrical stock about 4.5 inches in diameter and 4.5 inches long.

These samples cover at least five production lots of P-03 graphite, and therefore provide an indication of its average and minimum properties. In addition, the test results on P-03 plunger pins have been included to provide data on the actual hardware.

## EVALUATION PROCEDURES AND DATA

### PART I: PHYSICAL PROPERTIES OF P-03 GRAPHITE

#### Density Measurements

Graphite density was determined routinely from the weight-to-volume ratio for regularly shaped specimens such as flexure bars and compression cylinders. Density values provide a useful parameter for interpreting both the strength data and the structural quality of original stock. The individual density values will be routinely listed with their corresponding specimens. The following ranges were observed for various samples of P-03:

<u>STOCK TYPE</u>	<u>SAMPLE NUMBER</u>	<u>DENSITY (g/cc)</u>		
		<u>Low</u>	<u>Average</u>	<u>High</u>
Isost. molded	1	1.87	1.88	1.89
Isost. molded	2	1.82	1.84	1.87
Reg. molded	3	1.83	1.84	1.85
Reg. molded	4	1.82	1.85	1.86
ATJ		1.60	1.73	1.84

Density values for ATJ graphite (fabricated as a 9 x 20 x 24-inch molded block) have been given for comparison.<sup>6</sup> The spread in density for P-03 graphite is smaller than that of ATJ, while the average value is higher; this difference results both from the fabrication in small stock size and a considerably finer particle mix.

#### Graphitic Structure

One specimen of P-03 graphite from the first isostatically molded sample was compared to ATJ graphite by x-ray diffraction analysis. Specimen densities had previously been determined as 1.88 g/cc for P-03 and 1.75 g/cc for ATJ. The comparative analysis was made on a Siemens x-ray diffractometer by two methods: (1) measuring angular location of the intense x-ray line produced by diffracting the incident x-ray beam from the (002) basal planes in each graphite sample, and (2) measuring the shape of the two overlapping lines produced by

the x-rays diffracted from the (100) and (101) planes in each sample. The following results were obtained:

<u>PARAMETER</u>	<u>UNITS</u>	<u>P-03</u>	<u>ATJ</u>
$d_{(002)}$ spacing	Angstroms	3.37	3.36
Half-width of the (002) spacing	Degrees	0.41	0.40
$C_o$ parameter	Angstroms	6.74	6.72

In addition, it was noted that the (100) (101) band split was somewhat more pronounced in the ATJ sample, indicating a more ordered crystallite structure than found for the P-03 graphite.

The slightly broadened half-width of the (002) line could be due entirely to the smaller average particle size of P-03 as compared to ATJ. The higher value of  $d_{(002)}$  spacing for P-03 indicates a somewhat greater imperfection in its crystallite structure as compared with that of ATJ graphite; however, the ATJ is --in turn-- imperfect by comparison with the ideal 3.35 Angstrom value of this spacing for the theoretical graphite lattice. It is concluded that all of the given differences are small enough to be neglected, and P-03 may be regarded as a truly graphitic material.

Figure 2 presents a comparison between the microstructural features of P-03 and ATJ graphites. The smaller grain and pore size of P-03 are clearly evident.

### Radiographic Inspection of P-03

The first sample of isostatically molded P-03 was radiographed in an effort to define its internal structure. No voids or cracks were detected. One small inclusion (size about 1/32 inch) was found, corresponding to a well-defined inclusion of some unknown x-ray-opaque substance. Sample thickness was 2.0 inches.

In the case of regularly molded P-03 graphite twenty machined cylinders (size 4.0" D x 4.0" L) were inspected radiographically during this material's evaluation as an improved substitute for ATJ graphite. On the basis of this inspection. (both radial and axial exposures) fifteen of the twenty cylinders were accepted, and the remaining five rejected. The principal reason for the latter's rejection was the appearance of diffuse spots and streaks within the graphite, indicating the possible presence of low-density regions.

For the purposes of evaluation, the radiographic inspection of these cylinders was supplemented with the density measurements based on weight to volume ratio. The following results (including comparison tests on the hardware-grade ATJ graphite) were obtained:

<u>GRADE</u>	<u>NO. OF SAMPLES</u>	<u>RADIOGRAPHIC STATUS</u>	<u>DENSITY (g/cc)</u>		
			<u>Low</u>	<u>Average</u>	<u>High</u>
P-03	2	Accepted	1.847	1.851	1.855
P-03	5	Rejected	1.850	1.858	1.866
ATJ	12	Accepted	1.684	1.740	1.808
ATJ	12	Rejected	1.721	1.754	1.785

These results indicate that radiographically rejected samples are characterized by somewhat higher densities than those samples which are accepted. Since graphite strength tends to increase with density, the acceptance of low-density samples for hardware fabrication is a potentially undesirable trend for both the ATJ and P-03 structural graphites. The limited data obtained during this evaluation are only preliminary, but they indicate a need for monitoring the density of individual samples as part of the graphite quality control program.

In an effort to evaluate the strength of radiographically accepted versus rejected P-03 samples, one cylinder of each was chosen for extensive mechanical tests at room temperature:

<u>RADIOGRAPHIC STATUS</u>	<u>SERIAL NUMBER</u>	<u>AVERAGE DENSITY</u>
Accepted	22854	1.847 g/cc
Rejected	22850	1.850 g/cc

The choice of each cylinder was governed by the need for equal average densities and fairly large radiographic indications. Interpretation of the radiographic inspection results pointed to the presence of scattered regions of sub-average density, up to 0.4 x 0.5 inch in size. It was expected that in machining a large number of small specimens any adverse effect of these low-density regions would be readily demonstrated. However, the tests did not confirm the presence of such defects; the detailed results are given as part of the Discussion.

### Thermal Expansion

#### 1. WANL Measurements

Thermal expansion measurements were made on specimens radiantly heated in a helium atmosphere between 1100° and 2500°C (2100 to 4530°F). Specimen expansion was measured from the 2.00-inch reference length at room temperature. The lower temperature limit was imposed by the two-color optical pyrometer system for measuring the specimen's temperature; furnace temperature limitations imposed the upper limit.

The measured values of expansion were converted into thermal expansion coefficient by the conventional relationship

$$\bar{\alpha} = \frac{\Delta L}{L_0} \times \frac{1}{T - T_0}$$

where  $\bar{\alpha}$  is the linear coefficient of thermal expansion in a given grain direction, averaged between the room temperature ( $T_0$ ) and the test temperature ( $T$ ). Table 1 summarizes the results.

## 2. Other Measurements

Thermal expansion of the regularly molded P-03 graphite was determined for WANL by a commercial testing laboratory.<sup>7</sup> Specimens 5/16 inch in diameter and 4.00 inches long were heated in an electrical (wire-wound) resistance furnace. Expansion was measured by means of a modified optical strain-gage system. The resulting values are believed to be more accurate than the usual industrial measurements obtained with a dilatometer. Calculated values of the average thermal expansion coefficients are given in Table 1. Figure 3 summarizes these results graphically.

### Electrical Resistivity

Room-temperature measurements of electrical resistivity were made on selected flexure bars on a Kelvin Bridge (four-electrode) apparatus. The outer pair of electrodes, spaced three inches apart, served to hold the specimen. The inner pair of electrodes, spaced 2.5 inches apart, were pressed into contact with the specimen by a pneumatically-activated mechanism. This procedure minimized the contact effects; any measured variations in electrical resistivity would depend, therefore, on the material's structure.

Electrical resistivity was defined by the specimen geometry and resistance to electric current flow as follows:

$$R = r \times \frac{L}{A}$$

where R = electrical resistance of the flexure bar

L = inter-electrode span = 2.50 inches

A = specimen's cross section

r = electrical resistivity

Graphite resistivity is lower in the with-grain direction than across the grain due to the preferential orientation of the constituent particles that occurs during the conventional molding. Test results are summarized in Table 2.

Limited measurements of high-temperature electrical resistivity were made on one specimen in conjunction with the thermal conductivity determinations. Electrical resistivity values in Table 2 are corrected for the cross-section's change with thermal expansion. Figure 4 shows the variation of electrical resistivity with specimen density.

### Thermal Conductivity

High-temperature thermal conductivity was determined on one specimen machined from cylindrical regularly molded stock. This specimen was an electrically heated rod 0.50 inch in diameter and 4 inches long, equipped with threaded ends through which the electrical connections were secured. Its axis was oriented across the grain, hence providing heat flow radially in the with-grain directions. Temperature measurements were made with an optical pyrometer sighted on the surface and again into a small borehole; the latter was drilled to the specimen's center. The following values of thermal conductivity were obtained for the with-grain heat flow in P-03 graphite, together with comparison values for the ATJ graphite from the literature:

TEMPERATURE		THERMAL CONDUCTIVITY	
(°C)	(°F)	$\frac{\text{Cal-cm}}{\text{cm}^2 \text{ sec } ^\circ\text{C}}$	$\frac{\text{BTU-ft}}{\text{ft}^2 \text{ hr } ^\circ\text{F}}$
A. Results for the P-03 specimen (Sample 4-a)			
1100	2012	0.150	36.3
1600	2912	0.110	26.6
B. Comparison data for ATJ (literature, ref. 8)			
1100	2012	0.124	30.0
1600	2912	0.103	25.0

The thermal conductivity of P-03 is somewhat greater than that of ATJ graphite.

## PART II: MECHANICAL PROPERTIES OF P-03 GRAPHITE

### General

Conventional molding of graphite imparts an across-grain preferential orientation along the molding vector, together with the with-grain orientation perpendicular to this vector. Tensile strength is low when the stress and molding vectors coincide, and high when they are mutually perpendicular. For the compressive strength measurement, this relation is reversed.<sup>3</sup>

In the case of P-03 graphite, the above considerations are not entirely applicable. The size of constituent particles is smaller than that usually encountered in the conventional graphites, and the particles tend to be rounded. During a molding operation these small particles have less tendency to assume a preferential orientation than do large plate-like coke particles. The mechanical properties of P-03 consequently exhibit a greater uniformity and higher strength values when compared to the conventional molded grades.

### Tensile Strength and Elongation

Tensile properties of P-03 graphite were determined on a thread-loaded L-2 specimen (Figure 1-b) whose nominal gage section is 0.25 inch in diameter and 1.0 inch long. The total specimen length is 4.0 inches. (Ref. 1, Fig. 1). High-temperature tests were carried out on radiantly heated specimens in helium or argon atmospheres. Specimens were tested at a strain rate of 0.02 inch per minute. All fractures occurred either in the gage section (Figure 1-b) or at the point of the inner fillet's tangency.

The fracture behavior of L-2 specimens was noted to be consistent over a wide range of temperatures. Specimens tested at room temperature exhibited a single fracture if relatively weak; the strongest specimens broke with two fractures. In the 1000 to 2500°C (1830° to 4530°F) range, there were generally two fractures in a specimen, while at still

higher temperatures a single fracture was again observed. A similar behavior had been noted for the ATJ tensile tests. The second (and occasionally third) fracture is probably caused by the load train's backlash after the specimen's initial failure. Further aspects of this phenomenon have not been investigated.

The length of high-temperature specimens was measured with a micrometer before and after each tensile test in order to define the magnitude of permanent elongation. While graphite is a brittle material, it does, nevertheless, have a small capacity for undergoing a quasi-plastic permanent set in response to applied loads, in addition to the usual elastic strain. The latter disappears upon unloading the specimen, while the permanent set remains. Since the gage section is one inch long (plus a small length contributed by the tapered fillets), the measured elongation is numerically equal to the permanent strain in the specimen. At room temperature, the magnitude of permanent elongation did not exceed 0.001 inch, the lower limit of accuracy for the micrometer measurement technique used in this study.

Tables 3-a and 3-b summarize the results from all tensile tests. The first part covers isostatic P-03 graphite, while the second deals with the regularly molded stock. The identity of test stock samples has been preserved for the purposes of inter-comparison and statistical analysis of data. Figure 5 shows the variation of individual tensile strength values with temperature. The expected increase of strength with temperature is readily evident; at 2400°C the tensile strength of P-03 is nearly twice that at room temperature. Specimen elongation is given in Figure 6; it is seen to increase rapidly with temperature. Most of this elongation is confined to the gage section and the inner fillets; the approximate proportion is estimated in a later section.

### Preliminary Creep Tests

A limited number of high-temperature creep tests was made in tension on L-2 specimens as a supplement to the tensile test program. Each specimen was heated in a

helium atmosphere to the test temperature, and held for at least one hour to attain equilibrium. It was then stressed by a dead-weight loading arrangement; the initial stress was about 5% higher than the test stress (see the insert in Figure 7), in order to overcome any friction slip-page effects in the load train. Specimen length was measured before and after the creep test with a micrometer. In addition, the separation of two fiducial lines (engraved on the fillets) was determined with a toolmaker's microscope. Table 4 summarizes the creep test conditions. Figure 7 shows the specimen elongation under load as a function of time.

### Flexural Strength Tests - Circular Cross Section

Ten cylindrical rods, (0.374-inch diameter, 4.0 inches long with-grain orientation) were first tested in three-point loading, with 1.0-inch outer span. Their long fragments were then re-tested in loading, with a 1.0-inch inner span and 3.0-inch outer span. All these specimens were machined from regularly molded 4 x 4 x 1 1/4-inch plate stock. In addition, fifteen three-point tests were made in cut-down plunger pins machined from a different lot of the same size stock; these pins had been originally intended for reactor hardware, but were rejected for minor radiographic (x-ray) irregularities. All tests were made with a 0.02-inch per minute cross-head rate.

Table 5 summarizes the test results, while the individual values are plotted as a function of density in Figure 8. Flexural strength is seen to increase with density for both the three-point and four-point modes of loading.

In addition to the results listed in Table 5, twenty-seven regular plunger pins (rejected for minor radiographic irregularities) were tested in three-point loading. Their densities were not determined, and these results are hence excluded from this table; they are summarized below:

High	9640 psi
Average	8800 psi
Low	7450 psi
Extremely low	6070 psi

The last result was obtained on one pin with a readily visible surface defect, and is not regarded as being characteristic of the regular pins.

### Flexure Strength Tests - Square Cross Section

Flexure tests made on cylindrical specimens are not entirely suitable for evaluation purposes because the site of maximum tensile stress is confined to a small portion of the total surface. Since the initial fracture originates at this site, the apparent strength of a specimen will be influenced by any surface irregularities present in its vicinity. Specimens with square or rectangular cross section have a more uniform stress distribution, and reflect more nearly the quality of graphite. The majority of flexure tests intended for evaluation purposes were made on specimens having a square cross section.

Specimen size was generally  $7/16 \times 7/16 \times 3 \frac{1}{2}$  inches, but smaller bars were used occasionally where indicated. Specimens were loaded in a four-point test, generally with a 0.02 inch per minute platen motion rate. The loads were applied with rounded steel pins; for the regular test the pins were spaced 3.0 inches apart for the outer span (tensile face) and 1.0 inch apart for the inner span. This is called the third-point arrangement. Table 6-a summarizes these results for the isostatically molded P-03, while Table 6-b presents results for the regularly molded P-03. The distribution of individual test results with specimen density is shown in Figure 9, where the with-grain flexure strength of ATJ graphite has been added for comparison. It is seen that while the density of regularly molded P-03 is only slightly higher than that of ATJ (1.84 versus 1.72 g/cc), its flexure strength is nearly twice as large.

### Vibration Fatigue Test

Preliminary vibration fatigue tests were made in flexure on specimens machined from trimmed plate stock. These tests were conducted with a Baldwin-Hamilton model SF-2 constant load machine. Vibration frequency was 1800 cycles per minute. The specimens were clamped at one end between a pedestal and a top plate; the other end was bolted to a pivoted connecting

fixture which transmitted the sinusoidally varying bending load.<sup>(2)</sup>

The specimen's design (Figure 10, insert) was that provided by the manufacturer for testing sheet metal stock. The gage section was tapered to provide an equal stress distribution across its length; had the specimen been designed with a constant rectangular cross section, the peak stress would have been confined to the clamped end's base. The specimen was 0.25 inch thick, with an over-all length of 4 inches, and a maximum width of 2 inches.

The following results were obtained for the initial runs:

<u>SPECIMEN NO.</u>	<u>PEAK STRESS</u> (psi)	<u>LIFETIME</u> (cycles)	<u>FRACTURE SITE*</u> (inches)
1	5000	<10	1/4
2	3000	(0.233 × 10 <sup>6</sup> )	---
3	4000	(0.228 × 10 <sup>6</sup> )	---
4	4500	(0.201 × 10 <sup>6</sup> )	---
5	4800	(1.0 × 10 <sup>6</sup> )	---
6	5200	(1.032 × 10 <sup>6</sup> )	---
7	5000	6.749 × 10 <sup>6</sup>	3/8
8	5100	21.915 × 10 <sup>6</sup>	5/8

\*Distance from the base

The lifetime values in parentheses represent those tests which were arbitrarily terminated without achieving failure. The following results were obtained in the latter cases upon a subsequent re-run:

<u>SPECIMEN NO.</u>	<u>PEAK STRESS</u> (psi)	<u>LIFETIME</u> (cycles)	<u>FRACTURE SITE</u> (inches)
2	5400	<1	3/4
3	5300	<1	3/8
4	5100*	( $2.579 \times 10^6$ )	---
5	4800*	( $18.225 \times 10^6$ )	---
6	5200*	<10	1 1/8
4	5100*	<10	3/8

\*Re-test at previous stress level

All fractures occurred within the reduced section, between 1/4 and 1 1/8 inches from the base; the most common value was 3/8 inch. No fractures occurred at the minimal cross section located about 1 1/2 inches from the specimen's base.

Both the original and re-run fractures for specimens breaking below ten cycles were not expected. They may have been caused by transient overloads at the start-up; this seems probable because specimens No. 7 and No. 8 broke only after  $6.75 \times 10^6$  and  $21.9 \times 10^6$  cycles, respectively.

To obtain comparison data for the above tests, the base portion of fatigue specimens No. 1, 2, and 3 was sectioned into 0.25 x 0.50 x 2.0-inch flexure bars. For specimen No. 2, the bars were parallel to the vibration specimen's length, while for the other specimens, they were perpendicular; all, however, had with-grain orientation. The bars were tested at 0.2-inch/minute loading rate, with 0.5-inch inner span and 1.5-inch outer span. The following results were obtained:

<u>FATIGUE SPECIMEN NUMBER</u>	<u>DENSITY</u>	<u>FLEXURAL STRENGTH</u>
	(g/cc)	(psi)
1	1.835	8370
	1.840	7655
2	1.825	7920
	1.826	8110
	1.831	8075
3	1.846	7830
	1.848	8310
Average	1.837	8040
Std. Deviation	----	256

All results are summarized in Figure 10. Control flexure tests gave an average strength of 8040 psi, with a 256-psi standard deviation; the minimum expected flexure strength, defined as the average less three deviation values, is 7270 psi. The specimens broken in vibration had an average flexure strength (neglecting any transient overloading) of only 5155 psi, corresponding to a hypothetical stress concentration factor of 1.56.

#### Compression Tests

The compressive strength of P-03 graphite was determined on small cylindrical specimens of 0.5-inch diameter and 0.9-inch length. The load was applied by steel platens at a rate of 0.02 inch per minute. Specimen densities were determined from the weight/volume ratio as an auxiliary parameter. Table 7-a summarizes compression test data on the isostatically molded P-03 samples. Data for the regularly molded stock is presented in Table 7-b.

Figure 11 shows the individual test values for all compression specimens. For the regularly molded stock, the with-grain compressive strength is seen to be several

thousand psi greater than the across-grain strength. Results for the isostatically molded stock are intermediate, in excellent agreement. For comparison, the strength of ATJ graphite in compression is generally below 10,000 psi, in contrast to the 16,000 to 24,000 psi range for the P-03 graphite.

### Stress-Strain Curves for P-03

Room-temperature stress strain curves were determined for P-03 graphite by cementing resistance strain gages to typical tensile, flexural, and compression specimens. These specimens were loaded to destruction, with one or more intermediate load cycles imposed to define the stress-strain curve in greater detail.

#### Tensile Curves

Figure 12 shows the tensile stress-strain relationship for isostatically molded graphite. The curves were determined on a radially oriented L-2 specimen, machined from Sample No. 1; the specimen density was 1.88 g/cc. An insert in the figure demonstrates how the permanent set within the specimen increases with the preload.

Figure 13 presents the tensile stress-strain curve for regularly molded P-03, as determined on a with-grain specimen.

#### Compression Curves

A stress-strain curve in compression was determined on a typical cylindrical specimen machined from regularly molded graphite (Sample No. 4-d) in the axial orientation. Figure 14 presents the stress-strain curve for this specimen. An insert in the figure shows how the permanent set in this specimen varies with the peak stress.

#### Flexure Curves

To confirm the general shape of stress-strain curves in both tension and compression, one flexure specimen (machined axially from the second isostatic sample of P-03) was equipped with strain gages and tested to fracture. Specimen density was 1.840 g/cc. The test results are presented in Figures 15-a (tensile face) and 15-b (compressive face); they indicate that the

strains are linearly proportional to stresses at low loads.

#### Generalized Stress-Strain Curve

Figure 16 shows the general relationship of stress to strain for initial loading. It combines the pertinent data from Figures 12 through 15 with similar results determined on additional specimens. From this combined data it is seen that the secant Young's modulus of P-03 graphite is about  $2.0 \times 10^6$  psi at low stress levels; this modulus decreases to about  $1.5 \times 10^6$  psi near the fracture stresses. The tangent Young's modulus is equivalent to the secant modulus at low stress levels, but varies considerably more than the latter at high stress levels. Figure 16 is not applicable to the stress-strain behavior of pre-loaded specimens; in this instance, the detailed original curves should be considered.

The dependence of permanent set on the peak pre-load stress has been summarized in Figure 17. Permanent elongation is seen to be larger than the permanent contraction at identical pre-load stresses. The data from flexure tests are not in agreement with results of the pure tension and compression tests, for reasons presently undefined.

## DISCUSSION

### PART I: PHYSICAL PROPERTIES OF P-03 GRAPHITE

#### Density, Microstructure, and Strength

Mechanical properties of graphite are governed largely by its particle size, density, and the degree of anisotropy within the structure. Grade P-03 is particularly promising for structural applications because of its fine-grain microstructure, high density, and high strength in the generally weak across-grain direction.<sup>4</sup> High densities promote good surface finish, resistance to abrasion, and close dimensional tolerances on hardware components.

The advantages associated with both high density and isotropic microstructure are evident from the following comparison of three structural graphites:

<u>PARAMETER</u>	<u>UNITS</u>	<u>P-03</u>	<u>ATJ*</u>	<u>ZTA*</u>
Av. Density	g/cc	1.85	1.73	1.94
Tensile Strength				
With-grain	psi	4500	3355(380)	4400(670)
Across-grain	psi	4500	2935(120)	1530(300)
Compressive Strength				
With-grain	psi	21,000	8270(1030)	7100(1480)
Across-grain	psi	19,000	8540(1150)	12,600(1630)
Thermal Expansion (20 - 100°C)				
With-grain	°F <sup>-1</sup>	2.43	1.22	0.35
Across-grain	°F <sup>-1</sup>	2.88	1.90	5.0
Electrical Resistivity				
With-grain	milliohm-cm	1.75	1.10	0.70
Across-grain	milliohm-cm	1.95	1.45	2.2

\*Test results obtained from Ref. 9

It is evident that both P-03 and ATJ are relatively isotropic in their behavior, with P-03 graphite being the stronger grade. Both are molded graphites produced without reimpregnation

treatments. P-03 is also stronger than the dense grade ZTA; the latter, fabricated by a hot-molding process (with temperature about 2800°C, or 5070°F), is strongly anisotropic in both physical and mechanical properties. Both ATJ and ZTA grades are used to manufacture NERVA hardware, and on the basis of the above comparison, both may be replaced with P-03 graphite for improved performance, provided the other parameters are compatible with an intended application.

Figure 2 compares the microstructure of P-03 (regularly molded version) and ATJ graphite on photomicrographs taken at 100x magnification. The ATJ picture demonstrates the tendency of small coke grains to cluster into larger structures, thereby leaving large intergranular pores between the latter. This clustering tendency is not as pronounced in regularly molded P-03, which therefore retains the original fine-grained particles and has very small pores in comparison with ATJ. The higher strength of P-03 graphite is thus attributed to both its finer particle mix and small pore size.

### Electrical Resistivity

Resistance of graphite to the passage of electrical current provides a useful index of its microstructure and the degree of graphitization. Electrical resistivity is a factor which takes into account both the specimen's resistance and dimensions; this factor is therefore dependent only upon the structure for its magnitude. It is higher in the across-grain orientation than with the grain, and decreases somewhat with the higher graphitizing temperatures. Resistivity also decreases with higher bulk density since more conductive paths become available.

Figure 4 summarizes the variation of resistivity with density for both the regular and isostatic types of P-03. Most of the available data is for the 4.5"D x 4.5"L cylindrical stock. The following average values are obtained in this case:

<u>SAMPLE</u>	<u>ORIENTATION</u>	<u>SPECIMEN DENSITY</u> (g/cc)	<u>RESISTIVITY</u> (milliohm/cm)
4-c	WG	1.851	1.754
	AG	1.851	1.964
4-d	WG	1.849	1.762
	AG	1.850	1.980

The anisotropy in resistivity is about 12%, indicating that this stock is expected to have relatively uniform properties in all directions. Resistivity data for isostatically molded P-03 is not sufficient to find the anisotropy, but its value should not exceed that of the cylinder stock.

From Figure 4, it is evident that the electrical resistivity of P-03 plate stock (measured only in the with-grain direction) is considerably less than that of with-grain cylinder stock. This indicates that the plate stock has a more well defined grain orientation, and therefore greater anisotropy (as would be expected from the stock's large width/thickness ratio). Analysis of the resistivity versus density data leads to the relationship

$$\text{Log}_{10} r = -4.590 + 8.850 \times \frac{1}{d}$$

with  $r$  = electrical resistivity (milliohm-centimeters) and  $d$  = specimen density within the 1.80 to 1.86 g/cc range. The constants in this equation have been evaluated only for one lot of plate stock and may not be applicable for other lots. In view of the anisotropy differences between plates and cylinders, this formula does not apply to the latter.

Resistivity of P-03 graphite increases at cryogenic temperatures, but decreases to a minimum at elevated temperatures; this behavior conforms to that of other molded graphites. The following variation has been determined from preliminary data of Table 2:

<u>TEST TEMPERATURE</u>		<u>RATIO:</u>	<u>RESISTIVITY AT TEST TEMPERATURE</u>
(°C)	(°F)		<u>RESISTIVITY AT ROOM TEMPERATURE</u>
			(%)
-196	-320		143.5
30	86		100.0
23	73		100.0
1095	2003		60.8
1216	2220		60.5
1312	2394		60.9
1433	2610		61.0

### Thermal Expansion

Thermal expansion of graphite is considered here as an averaged coefficient of linear expansion between the room and test temperatures. This coefficient tends to increase with test temperature, and is always higher in the across-grain direction than with the grain. Its magnitude is generally lower for graphites than for other structural materials; this complicates the problem of attachment to non-graphite materials by creating interface stresses.<sup>3</sup> For P-03 however, the expansion coefficient value is greater than for the regular graphites, and the interface stresses would tend to be somewhat decreased.

Thermal expansion coefficient is shown in Figure 3 as a function of test temperature for both regular and isostatically molded graphites. For isostatic P-03, the expansion at high temperatures (up to 2480°C or 4500°F) is nearly independent of orientation; the anisotropy is less than 10%. The data from each isostatic sample are in excellent agreement.

In the case of regularly molded P-03, the expansion measurements on one with-grain and one across-grain specimen machined from cylindrical stock show the anisotropy of about 20%. For plate stock, only the with-grain measurements are available, but the anisotropy must be higher than that of cylinders due to the more pronounced grain alignment within the plates.

Thermal expansion in the with-grain direction is nearly equal for specimens machined from both large (12 x 12 x 1.2 inches) and small (4 x 4 x 1.1 inches) plate stock; the values are smaller than those for cylindrical stock.

It should be noted that high temperature and intermediate-temperature measurements of thermal expansion (Figure 3) were made with different equipment and on different stock. The high-temperature test results are available only for isostatic P-03 graphite, while the regularly molded P-03 has been tested entirely in the intermediate range. Test results for the regular stock are considered more reliable than those for the isostatic stock because equipment calibration at the intermediate temperatures is superior to that available at high temperatures.

## PART II: MECHANICAL PROPERTIES OF P-03 GRAPHITE

### General

Mechanical properties of P-03 were briefly treated in the earlier discussion. Comparison of this grade with ATJ and ZTA graphites showed the advantages associated with the high density and low anisotropy of P-03. This section will cover mechanical properties in greater detail, with an emphasis on the regularly molded material.

### Tensile Strength - Room Temperature

#### Regularly Molded Stock

The tensile strength of regularly molded cylindrical stock has been adequately investigated in both the with-grain (radial) and across-grain (axial) orientations. Test results have been summarized in Table 3-b. The anisotropy between these two directions is less than the magnitude of one standard deviation. Their tensile strength values are combined for increased numerical sampling, assuming the strength of cylindrical P-03 stock is nearly independent of orientation. The following results are then obtained, in the order of increasing stress at fracture:

<u>Cumulative Rank*</u>	<u>Tensile Strength (psi)</u>	<u>Orientation</u>		<u>Cumulative Rank*</u>	<u>Tensile Strength (psi)</u>	<u>Orientation</u>	
		<u>WG</u>	<u>AG</u>			<u>WG</u>	<u>AG</u>
1	3700	X		11	4700	X	
2	3790	X		12	4810		X
3	4070	X		13	4870	X	
4	4100	X		14	4890		X
5	4100	X		15	4930	X	
6	4400	X		16	5000	X	
7	4300	X		17	5090	X	
8	4460		X	18	5570	X	
9	4470	X		19	5640		X
10	4540		X	20	5740		X

Average = 4680; Std. deviation = 564

\*By failure

These data for twenty-one specimens tested at room temperature give a tensile strength that varies between 3700 and 5740 psi, with a median at 4790 psi.

The across-grain specimens tend to be stronger than the with-grain specimens, which is not generally the case for the usually well-oriented molded graphites. This difference is real, and cannot be attributed to improper identification of the tensile specimens. Measurements of electrical resistance on unbroken specimens confirm their orientations:

<u>SAMPLE</u>	<u>ORIENTATION</u>	<u>RESISTANCE</u> (milliohms)	<u>TENSILE STRENGTH</u> (psi)
4-c	WG	23.3	4070, 4790, 5090
		23.5	4100
		23.7	3700
4-c	AG	28.9	4890, 4890
4-d	WG	23.3	4400, 4430, 4930
		23.5	3790, 4100
4-d	AG	26.7	4460, 4540

Specimen resistance was measured with a 2.5-inch span centered on the shoulders immediately below the threads.

The higher tensile strength of across-grain specimens is evidently due to both the fine-grained particles and a rather large shrinkage of the cured mix during the subsequent bake treatments.<sup>10</sup> This shrinkage compensates for much of the preferential particle orientation established when the mix is first molded. The shrinkage is beneficial, however, in achieving the high final packing density necessary to attain the high strength of the fully graphitized material. The relatively isotropic physical properties of cylindrical stock are thus possibly explained by the de-orientation effects of shrinkage.

In the case of regularly molded P-03 plate stock, the with-grain tensile strength was 6390 psi (one specimen); this was the highest value ever obtained after many tests on both P-03 and ATJ graphites.<sup>6, 9</sup> The plate stock was not thick enough for across-grain tests; however, the pronounced orientation effects and the high with-grain strength suggest the values of 4000 to 5000 psi for the across-grain tensile strength of the plate stock.

#### Isostatically Molded Stock

Six tensile tests were made at room temperature on isostatic stock. Their results fall entirely within the tensile strength range of regularly molded cylindrical stock. The first sample of isostatic stock was stronger than the second sample, due to the differences in density:

<u>SAMPLE</u>	<u>ORIENTATION</u>	<u>DENSITY</u> (g/cc)	<u>TENSILE STRENGTH</u> (psi)
No. 1	Radial	1.88	5130, 5680
No. 2	Axial	1.84	4080, 4140, 4280, 4400

It is evident that the isostatic molding process for P-03 graphite results in an excellent structural material.

#### Tensile Strength and Elongation at High Temperature

High-temperature tests on P-03 graphites were made on specimens from both the isostatic (Sample No. 2 only) and regularly molded (cylindrical) stock. The specimens were tested at several temperatures in the 1000° to 2800°C (1830° to 5070°F) range. The results (Figure 5) show that tensile strength of both the regular and isostatically molded P-03 graphites increased with temperature to a maximum at about 2400°C (4350°F); at this maximum, the tensile strength was nearly 100% greater than at room temperature.

Although classed as a brittle material, graphite does have a finite elongation before fracture; the magnitude of this elongation increases rapidly with temperature, increasing by a factor of about 100 between room temperature and 2800°C (5070°F), as shown in Figure 6.

Specimen elongation was determined by three techniques. The simplest method was based on measuring the length of L-2 specimens with a micrometer before and after the test. At room temperature, this difference did not exceed 0.001 inch (one mil), but at high temperatures it was as high as 0.030 inch. This method was not suitable for those specimens which broke into three or more fragments. A comparable method involved the measurement of separation between two fiducial marks with a toolmaker's microscope; the marks were placed on the inner fillets, about 0.22 inch above the point of fillet tangency, in order to avoid their influence as local stress-raisers. This second method was perhaps ten times more accurate than the first. Finally, the room-temperature strains were measured directly by means of a strain gage, and resulting elongation in the gage length determined directly; this sensitive method permitted the elongation to be determined both before and after the fracture, and thereby define the magnitude of both elastic and permanent strain in graphite.

In the case of creep tests, the elongations were measured both by the micrometer and toolmaker's microscope methods. These data are compared below:

MICROMETER DATA		MICROSCOPE DATA		CALCULATIONS	
<u>Specimen Length</u>	<u>Specimen Elongation</u>	<u>Section Length</u>	<u>Sectional Elongation</u>	<u>Gage Length</u>	<u>Gage Elongation</u>
(in.)	(mils)	(in.)	(mils)	(in.)	(mils)
4.0	3	1.42	2.2	1.0	1.7
4.0	7	1.45	6.1	1.0	4.6 <sub>3</sub>
4.0	9	1.43	5.4	1.0	4.1 <sub>5</sub>

This comparison shows that only about 55% of the unbroken specimen's elongation occurs within the gage section. The permanent strain to fracture is thus numerically equal to about one-half of the broken specimen's elongation.

### Flexural Strength of P-03 Graphite

Flexure tests were made on specimens machined with both square and circular cross sections. Specimens with the square cross-section give values that are characteristic of the material's properties. The round specimens, on the other hand, are similar to reactor hardware (plunger pins), and are thus suitable for quality control testing. The test results for round bars are given in Table 5, and for square bars in Table 6. The use of more complex shapes for flexure tests on graphite is ruled out by the extra machining effort, with only marginal improvement in the results.

Interpretation of flexure data for both the round and square bars involves a fundamental assumption that these tests were made successively on the same specimen of reasonably uniform density. Three-point tests give an average flexure strength of 9490 psi, with a standard deviation for ten tests of 1320 psi or 13.9% of the average. Four-point tests gave 8080 psi average, with 970 psi (12.0%) deviation. Specimens with a square cross section but equal loading spans gave 8215 psi (average of 2 tests); this average value is about 500 psi lower than the flexure strength results obtained on round specimens of the same density. It is hence inferred that the four-point flexure strength of square cross section specimens would be about 500 psi less than the results from round specimens, although this comparison needs confirmation.

Previous work on ATJ molded graphite had shown that the failure of square flexure bars is governed primarily by irregularities on the tension surface.<sup>6</sup> P-03 graphite should be governed by the same factor. None of the test specimens exhibited cracks or chipped-out regions, and the breakage rate in machining was negligibly low. After testing, all fractures

were found to be limited to the inner span, where the tensile-face stress is a maximum for any given load. All of these considerations show that P-03 graphite has a relatively defect-free structure.

Figure 9 compares the flexure strength of P-03 and ATJ on the basis of density. ATJ values are based on 18 four-point flexure tests for the regular specimens machined in the with-grain (strongest) direction; across-grain average strength would be about 600 psi lower.<sup>6</sup> The advantage of high density is readily demonstrated for P-03 graphite, since its strength is at least 50% higher than that of ATJ.

#### Bending Fatigue Strength of P-03 Graphite

Fatigue behavior of graphite has not been extensively investigated, although the influence of alternating stresses is expected to reduce the value of minimum design load in much the same manner as for cast iron. Preliminary measurements were therefore undertaken on P-03 graphite as a part of this evaluation; the tests were limited to with-grain specimens machined from plate stock. The experimental procedure was described earlier; test results are summarized in Figure 10.

The fatigue specimens broke at 5155 psi average flexural strength, in contrast to the control flexure strength of 8040 psi. Specimen failures occurred either upon the initial loading, or after several million cycles of fatigue exposure. There was no evidence for the progressive decrease of strength with exposure to vibration, such as found recently for the molded fiber-base graphites.<sup>2</sup> The P-03 specimens that failed during the initial loading may have been subjected to the start-up overloads; they are assumed to have failed during the first quarter of the loading cycle when surface stress in the gage section is at its maximum value. The experimental technique was such that initial failures could have occurred between 1/4 and 5 cycles; the fatigue machine's pre-set frequency of 1800 cycles per minute made it difficult to follow the test visually.

Once past the initial loading, some specimens underwent a million cycles without a fracture. However, upon a re-start at the previously safe or even lower peak stress, many of the surviving specimens broke again during the initial loading. All fractures were confined to the gage section, regardless of whether the specimen broke initially or after several million cycles. Comparison of all fatigue results to control flexure tests indicates that the former may be affected by a hypothetical stress concentration factor of 1.56. The nature of this factor is not clear at this time.

### Compression Strength of P-03 Graphite

Compression test results on P-03 (given in Table 7 and summarized in Figure 11) are in the  $20,000 \pm 3000$  psi range. All tests were made on small cylindrical specimens 0.50 inch in diameter and 0.90 inch long. Examination of the crushed specimens indicated that the original fractures were located within about 1/4 inch of one of the loaded faces. These fractures formed a V-shaped wedge pointing into the specimen. The subsequent fractures were due first to the disruptive influence of the wedge, and later to the buckling-type failure of the axially oriented secondary fragments.

The mechanism of failure in a compressively loaded graphite cylinder is deduced to be the following:

#### Stage 1: Original Failure

This is the least known aspect of the failure mechanism. The disrupted surface and characteristic geometry of the wedge-shaped fragment (Stage 2) indicate that initial failure in the specimen may be governed by a shear-dependent mechanism. It may be mentioned that compression tests on well-oriented extruded graphites (compressive strength about 9000 psi) tend to produce a clean fracture with a single surface, as if only one-half of the wedge were present.

### Stage 2: Wedge Formation

Following the original failure near one of the loaded faces, a wedge is rapidly formed. Post-fracture examination of this wedge shows that its faces have a dark powdery appearance (Figure 1-b) as if disrupted by sliding under a severe load. The wedge points toward the undisrupted end of the specimen. Compression strength values probably reflect the nominal stress at which this wedge is formed, since they are based on the first discontinuity in the load-versus-deflection curve.

### Stage 3: Wedge Splitting

Upon continued loading, the wedge splits the specimen into two or more axial fragments. The interface surfaces between such fragments have distinctly different appearance than those associated with the wedge, and resemble the fracture surface of tensile or flexural specimens.

### Stage 4: Buckling

Continued application of the load causes the irregular axial fragments to fail by buckling. Further loading would, of course, simply break up the various larger fragments. Compression tests were rarely taken to the buckling stage.

Pre-fracture behavior of loaded P-03 cylinders is defined by the strain-gage data in Figures 16 and 17. Initial loading produces a linear stress-strain curve to perhaps 10,000 psi. Upon continued loading, the structure becomes less rigid, possibly due to a disruption or realignment of binder bridges between the particles; the slope of the stress-versus-strain curve then decreases somewhat. Above about the 15,000 psi level, graphite seems to regain its rigidity, probably due to the closing up of the smaller pores within the structure. Near 20,000 psi stress levels the specimen fails, undergoing the previously described fracture sequence.

## Fracture Statistics

Being porous and brittle materials, all graphites exhibit a large variability in their mechanical properties. Grade P-03 is expected to show some of this variability, particularly when the results of a given test on the reasonably homogeneous material are considered for a large number of specimens.

### Reasons for Strength Variation

The strength of graphite is influenced by the following factors:

1. Microstructural details (particle type, size, shape; binder bridge strength; pore geometry and fraction).
2. Structural defects (voids, cracks, regions of higher and/or lower than average density, high ash areas).
3. Packing density (specimen density after graphitization).
4. Preferential orientation of the particles (with-grain and across-grain anisotropy).
5. Degree of graphitization.

Further factors (in particular, neutron irradiation) cannot be considered at this time because of the absence of data.

Being a premium grade of graphite, P-03 is produced with practically no gross structural defects; it has a relatively uniform degree of graphitization, and a uniformly high packing density. Due to the small particles predominant in the original mix, and the shrinkage effects during initial carbonization of the binder, the preferential orientation in the graphitized material may be neglected for cylindrical raw-stock. Thus, only the microstructural details remain as the major variable influencing the strength of P-03 graphite.

Evaluation tests on P-03 have not been numerous. However, its uniform density and relatively isotropic properties permit the data for several stock batches and all orientations to be compared together for any strength tests. In this way the test results of about fifty specimens may be obtained, thereby providing good numerical indications for the low, average, and high strength results.

Statistical presentation of combined strength data was based on the arrangement of test results in the order of increasing strength. The isostatically molded P-03 stock was assumed equivalent to regularly molded cylindrical stock, provided the densities of the two were equal. Sample No. 1 of the isostatic P-03 and the plate stock are treated separately; the high density of the former, and relatively pronounced orientation in the latter caused their mechanical results to be outside the scatter range of the other P-03 stock.

Reactor plunger pins have been extensively tested for quality control purposes in the previously described three-point loading arrangement. Figure 18 shows the cumulative number of broken pins, plotted as a function of the peak stress on the pin's tensile surface. These results reflect the high with-grain strength of the P-03 plate stock, from which all the pins had been machined.

Examination of Figure 18 shows that the weakest fractures occurred near 6000 psi for two specimens. One of these was a plunger pin rejected during visual inspection because of a crack on its surface. The other low-strength specimen appeared to be free of cracks, but had an unusually low density and an irregular surface finish. The weakest of the remaining specimens broke above 7000 psi. The average three-point flexure strength for all tests was 8820 psi, while the median fell slightly lower to 8765 psi; the 55 psi difference is well below the magnitude of one standard deviation.

## Tensile Strength

Figure 19 shows the cumulative distribution of tensile strength for twenty specimens machined in both the with-grain and across-grain orientation from regularly molded cylindrical stock.

Fracture probability is assumed to be proportional to the cumulative rank by failure, with 100% probability corresponding to the maximum observed tensile strength of 5740 psi. The resulting plot of applied tensile stress versus failure probability for the regularly molded P-03 cylindrical stock is shown in Figure 19. These tensile results are in excellent qualitative agreement with three-point flexure tests on plunger pins; in both cases the data tend to cluster into "families" with low dispersion, separated by relatively large dispersion between them. The reason for such clustering is not clear. If several types of stress-raisers are assumed to exist in graphite, with each stress-raiser active at a particular stress level, the results of Figures 18 and 19 become reasonable. Specimens breaking at low stresses have the most effective stress-raisers, while the strongest specimens probably represent relatively flaw-free graphite.

## Flexure Strength

Figure 20 gives the cumulative strength distribution for the four-point flexure test on the square bar-shaped specimen. This may be compared to the three-point flexure test on round specimens (Figure 18). Departure of the data in Figure 20 from symmetrical distribution at high stresses may be readily traced to the inhomogeneous specimen population; the highest strength is exhibited by specimens from the first isostatic sample (density too high compared to the majority of other specimens) and the regularly molded plate stock (highly developed orientation). Figure 20 may therefore serve as an example of results attained with a mixed population.

## Compressive Strength

Results of compression tests on P-03 graphite of comparable density and grain orientation are shown in Figure 21. The form of their distribution is similar to those for the tensile and compressive strength results.

## X-ray Inspection of P-03 Graphite

In the course of radiographic inspection of rough-machined P-03 blanks (4.0"D x 4.0"L) it was noted that rejected pieces tended to be of higher density than those accepted. The same situation was found for ATJ graphite blanks. The details were given previously; here only a brief comparison of the accepted versus rejected P-03 will be presented, as determined on one blank of each. The results should not be regarded as conclusive at this stage.

First, the local densities of one accepted and one rejected cylinder were investigated by evaluating them in flexure and compression. Tensile tests were also made on both cylinders, but the density of L-2 specimens could not be determined. All specimens were machined in an identical manner from both cylinders. The following density values were obtained:

Stock Number	X-Ray Status	Cylinder Density	Specimen	Density		
				Low	Avg.	High
22850 (Sample 4-c)	Rejected	1.850	Compression	1.844	1.850	1.853
			Flexure, axial	1.847	1.850	1.854
			Flexure, radial	1.849	1.850	1.853
22854 (Sample 4-d)	Accepted	1.847	Compression	1.833	1.846	1.864
			Flexure, axial	1.848	1.849	1.852
			Flexure, radial	1.848	1.849	1.852

Although cylinder No. 22850 was rejected for low-density indications, its density variations are less than those in the accepted cylinder No. 22854. The lower average density and the greater density spread for the latter cylinder were subsequently reflected in the lower flexural and compressive strength values, and increased standard deviations.

These results are not numerically significant for a reliable correlation of x-ray inspection techniques with graphite strength. However, several generalizations are possible, particularly since the P-03 plunger pins are routinely inspected by radiography, and their rejection rate has been high. First, the excellent structure of P-03 was reflected on all radiographs examined from three lots of plunger pins, one lot of rough-machined cylinders, and one sample of isostatically molded graphite. The absence of cracks, voids, chipped-out regions, and similar defects on the surface of all evaluation specimens agrees with the observation of not more than five surface cracks in over one thousand hardware pieces; all this evidence for the defect-free macrostructure of P-03 was reflected by the absence of well-defined radiographic defects. Only one high-density inclusion had been detected; it occurred in the isostatic sample. Radiographic defects attributed to P-03 graphite's structure were noted to be minor irregularities with diffuse boundaries and often inadequate definition; any correspondence with the actual features of P-03 structure are questionable.

Radiography of graphite is one of the more controversial subjects concerning this material, and there is disagreement on whether x-ray inspection offers a suitable procedure for monitoring either strength or structure, particularly for the purpose of enhancing the hardware reliability. Each application should be considered separately, and the previous experience with a premium fine-grained grade such as P-03 would offer no guidance where other types of graphite are concerned.

#### Predicting the Strength of P-03 Graphite

To secure the best structural graphite hardware for a reactor, it is necessary to select the graphite stock with uniformly high strength. Since strength increases with density, the raw-stock may be monitored to reject low-density pieces; for best results, density should be determined on the rough-machined blanks rather than on the original stock. Density measurements may also be made on the finish-machined hardware of simple geometry (such as the

plunger pins) but in this instance any rejection is necessarily more expensive than for the rough-cut blanks.

Another way of predicting adequate strength of graphite in the early stages of hardware procurement is to measure the graphite's hardness. This measurement may be performed through either the re-bound or indentation techniques. Neither of these techniques has yet been tried at WANL on P-03 graphite, although the manufacturer used a re-bound technique for the regular quality control testing.<sup>10</sup> Indentation technique has been tried on ATJ molded graphite, and an excellent correlation between the hardness numbers and flexure strength secured.<sup>6</sup> Quality control by means of hardness testing is perhaps the most promising technique, since it may be performed directly on raw-stock.

Electrical resistivity measurements may be made on simple, axially symmetrical shapes such as plunger pins. This technique has been tested on hardware-grade plate stock. Figure 22 shows the resistivity versus flexure strength correlation for P-03. At a constant value of resistivity, the flexure strength varies somewhat, due most likely to the influence of surface irregularities. Four-point test values give a better correlation with resistivity than do the three-point results. In the case of ATJ graphite, the resistivity of flexure bars was found to be inversely proportional to average flexure strength when both were measured on specimens with a square cross section.

## CONCLUSIONS

1. Mechanical properties of the premium molded P-03 grade of graphite have been found to be the highest among those structural grades for NERVA hardware.
2. After testing a large number of specimens in tension, flexure, and compression, the regularly molded P-03 graphite is found to have both a high average strength and low standard deviation.
3. Isostatically molded P-03 graphite, available presently on a semi-commercial basis, is found to be either equivalent or superior to regularly molded stock.
4. The isostatic version of P-03 has uniformly high mechanical properties in all orientations; it may be considered therefore as a strong, uniform, isotropic, structural material.
5. Regularly molded cylindrical stock (original size 4.5"D x 4.5"L) is comparable to the isostatic P-03 in most of its properties. The major difference is that the isostatic version may be produced in larger size, both diameter and length.
6. Regularly molded P-03 plate stock has a more highly developed orientation than cylindrical stock. Its properties are therefore expected to be moderately anisotropic.
7. All versions of P-03 have uniformly high density, generally in the  $1.85 \pm 0.03$  g/cc range. They are resistant to abrasion and indentation and have a good surface finish when machined.
8. Being a dense molded graphite, P-03 has been found to be remarkably free from internal defects from visual examination of the finish-machined surfaces and radiographic inspection.

9. In addition to mechanical tests, selected physical and thermal properties of P-03 have been studied. These results have been covered in this report in sufficient detail to allow preliminary designs to utilize this material.

10. Radiographic inspection of P-03 graphite at a 2% sensitivity level does not provide a reliable indication of its macrostructural quality.

11. Extension of grade P-03 from its present applications to the other graphite components will result in significantly improved hardware.

## APPENDIX I: P-03 SUPPORT BLOCKS

Four support blocks machined from regularly molded P-03 have been tested by the Engineering Mechanics Department. These blocks showed excellent performance under the conditions of simulated reactor loading at room temperature. The catastrophic failures previously associated with ATJ graphite support blocks near the 4000-lb. load were not encountered; instead, the fractures in the simulated hardware assembly occurred outside the P-03 blocks at higher loading. A load of 8500 lbs. was found necessary to fracture a P-03 support block in an all-metal test fixture; this is estimated to be approximately 300% higher than the tensile loading that could be safely sustained by a tie rod at room temperature. The preliminary test results demonstrate that P-03 support blocks may be expected to carry design loads about 100% greater than those used for the present blocks machined from ATJ graphite.

### ACKNOWLEDGEMENTS

Special thanks are due to Dr. Robert R. Paxton, Chief Engineer of Pure Carbon Company, for his many valuable comments on the manufacture and properties of P-03 graphite, and for providing samples of the isostatically molded P-03 for this evaluation.

Within the Materials Department, the writer wishes to thank A. W. Danko for an x-ray diffraction analysis; P. S. Gaal for most of the thermophysical data; and E. F. Vandergrift for stress-strain curves. Radiographic quality of P-03 graphite was determined with the cooperation of J. R. Steele, Supervisor of the Non-Destructive Test Laboratory, Quality Control Department.

TABLE I

 SUMMARY OF THERMAL EXPANSION MEASUREMENTS FOR P-03 GRAPHITE THERMAL EXPANSION  
 COEFFICIENT, IN MICROINCHES PER INCH PER °F

TEMPERATURE (°C)	(°F)	ISOSTATICALLY MOLDED STOCK			REGULARLY MOLDED STOCK			
		SAMPLE 1 RADIAL	AXIAL	RADIAL AXIAL	SAMPLE 3 W.G.*	W.G.**	W.G.	A.G.
20	68	---	---	---	---	---	---	---
100	212	---	---	---	1.755	1.72	2.43	2.88
200	392	---	---	---	1.885	1.80	2.43	2.92
300	572	---	---	---	1.965	1.92	2.46	3.01
400	752	---	---	---	2.02	2.04	2.53	3.07
500	932	---	---	---	2.05	2.14	2.59	3.10
600	1112	---	---	---	2.13	2.22	2.62	3.15
700	1292	---	---	---	2.21	2.28	2.64	3.18
800	1472	---	---	---	2.27	2.33	2.67	3.21
1093	2000	2.33	2.46	2.24	---	---	---	---
1371	2500	2.38	2.51	2.43	---	---	---	---
1649	3000	2.52	2.66	2.63	---	---	---	---
1923	3500	2.74	2.86	2.85	---	---	---	---
2205	4000	2.92	3.02	3.06	---	---	---	---
2484	4500	3.07	3.15	3.37	---	---	---	---

Specimen Densities (g/cc)

1.875    1.870    1.84    1.84    1.77    1.84    1.84    1.86

\*Measured on the 4 x 4 x 1.1-inch stock

\*\*Measured on the 12 x 12 x 1.2-inch stock

TABLE 2  
ELECTRICAL RESISTIVITY OF P-03 GRAPHITE

<u>ORIENTATION</u>	<u>DENSITY</u> (g/cc)	<u>TEMPERATURE</u>		<u>RESISTIVITY</u> (milliohm - cm)
		(°C)	(°F)	
A. Isostatically molded graphite, Sample 1				
RADIAL	1.87	30	86	1.79
	1.87	-196	-320	2.57
B. Isostatically molded graphite, Sample 2				
AXIAL	1.840	23.5	74	2.12
	1.842	23.5	74	2.10
	1.845	23.5	74	2.08
C. Regularly molded graphite, plate stock, Sample 3				
No tests				
D. Regularly molded graphite, cylindrical stock, Sample 4-a				
A. G.	1.85	23	73	1.960
	1.85	1095	2003	1.191
	1.85	1216	2221	1.186
	1.85	1312	2394	1.193
	1.85	1433	2611	1.195
E. Regularly molded graphite, cylindrical stock, Sample 4-c				
W. G.	1.849	23	73	1.755; 1.760
	1.851	23	73	1.740; 1.760
	1.852	23	73	1.760
	1.853	23	73	1.740
A. G.	1.847	23	73	1.980
	1.850	23	73	1.945; 1.990
	1.851	23	73	1.930; 1.960
	1.854	23	73	1.980
F. Regularly molded graphite, cylindrical stock, Sample 4-d				
W. G.	1.839	23	73	1.760
	1.845	23	73	1.790
	1.849	23	73	1.760
	1.852	23	73	1.740; 1.775
	1.855	23	73	1.740
A. G.	1.848	23	73	1.980; 1.990; 2.010
	1.851	23	73	1.960; 1.990
	1.852	23	73	1.945
G. Tests on hardware samples -- regularly molded stock				
W. G.	1.809	23	73	1.990
	1.821	23	73	1.840
	1.832	23	73	1.745; 1.800
	1.843	23	73	1.615
	1.845	23	73	1.615
	1.853	23	73	1.530; 1.550
H. Comparison data for ATJ graphite				
W. G.	1.753	23	73	1.090
	1.744	23	73	1.512
A. G.	1.786	30	73	1.050
	1.786	-196	-320	1.740

**TABLE 3-A**  
**TENSILE TEST RESULTS ON ISOSTATICALLY MOLDED P-03 GRAPHITE**

ORIENTATION (1)	TEST TEMPERATURE		TENSILE STRENGTH (psi)	ELONGATION (2)		NO. OF FRACTURES (3)
	(°C)	(°F)		(mils)	(%)	
<b>A. SAMPLE NO. 1</b>						
RADIAL	RT	RT	5131, 5683	< 1	< 0.1	-
<b>B. SAMPLE NO. 2</b>						
AXIAL	RT	RT	4080, 4140, 4290, 4400	< 1	< 0.1	1 or 2
	1000	1830	5050	< 1	< 0.1	
	1400	2550	6330	< 1	< 0.1	2
	1800	3270	6390	< 1	< 0.1	2
	2000	3630	6800	< 1	< 0.1	2
	2200	3990	7950	2.5	0.25	3
	2400	4350	8450	10.5	1.05	1
	2500	4530	7440	11.7	1.17	1
	2600	4710	6420	18	1.8	1
	2800	4070	5180	33	3.3	1

(1) Orientations are either axial (parallel to the pressing's axis) or radial; the latter designation includes those specimens parallel to a radius but not oriented toward the center.

(2) Elongation is the difference in specimen length before and after the test, as determined with a micrometer.

(3) Percentage values of elongation are derived from the actual values by assuming that all deformation occurs in the 1.0-inch gage section. These values should therefore be regarded as the upper limits.

TABLE 3-B

## TENSILE TEST RESULTS OBTAINED ON THE REGULARLY MOLDED P-03 GRAPHITE

ORIENTATION AND IDENTITY (1)	TEST TEMPERATURE		TENSILE STRENGTH (psi)	ELONGATION		NO. OF FRACTURES
	(°C)	(°F)		(mils)	(%)	
A. Sample 3, Plate stock (trimmed, 2 x 6 x 1 1/4-inch size)						
W. G.	RT	RT	6390	< 1	< 0.1	---
B. Cylindrical stock, 4.5"D, 4.5"L						
W. G. 4-a	RT	RT	4470, 4870	< 1	< 0.1	1,1
	RT	RT	5000	< 1	< 0.1	2
W. G. 4-b	RT	RT	5570	< 1	< 0.1	2
W. G. 4-c	RT	RT	3700, 4070, 4100	---	---	all 1
	RT	RT	4790, 5090	---	---	all 1
W. G. 4-d	RT	RT	3790, 4100, 4400	---	---	all 1
			4430, 4930	---	---	all 1
W. G. 4-a	1200	2190	4690	---	---	all 1
W. G. 4-b	1200	2190	6030	3	0.3	2
W. G. 4-a	1500	2730	6520	< 1	< 0.1	3
W. G. 4-u	2000	3630	6680	---	---	3
W. G. 4-b	2000	3630	8070	---	---	2
W. G. 4-a	2400	4350	8780	9	0.9	5
W. G. 4-a	2600	4710	7620	19	1.9	1
A. G. 4-2	RT	RT	5640	< 1	< 0.1	1
A. G. 4-b	RT	RT	4810, 5740	< 1	< 0.1	1,1
A. G. 4-c	RT	RT	4890, 4890	< 1	< 0.1	1,1
A. G. 4-d	RT	RT	4460, 4540	< 1	< 0.1	1,1
A. G. 4-b	1200	2190	5940	< 1	< 0.1	2
A. G. 4-a	1500	2730	6900	< 1	< 0.1	1
A. G. 4-b	1500	2730	6990, 7250	< 1, 1	< 0.1; 0.1	2, 2
A. G. 4-b	2000	3630	8640	< 1	< 0.1	2
A. G. 4-b	2400	4350	9020	3	0.3	2
A. G. 4-a	2600	4710	7650	---	---	1
A. G. 4-b	2600	4710	8540 <sup>(2)</sup>	7	0.7	2

NOTES: 1. Letters a and b refer to particular items of stock for identification purposes.

2. Strain rate 0.2 inch per minute, compared to 0.02 for the other tests.

**TABLE 4**  
**SUMMARY OF CREEP TEST DATA**

ORIENTATION	TEST TEMPERATURE		TEST CONDITIONS		SPECIMEN ELONGATION (1)	PERMANENT STRAIN (2)	RE-TEST STRENGTH	CONTROL STRENGTH
	(°C)	(°F)	STRESS (psi)	TIME (min.)				
<b>A. Isostatically molded P-03 graphite; Sample 2</b>								
AXIAL	2600	4710	4050	5.0	9	3.78	4570 (3)	4225
<b>B. Regularly molded P-03 graphite; Sample 4-a (cylindrical stock)</b>								
W. G.	2600	4710	4070	5.0	7	4.20	3800 (3)	4780
W. G.	2000	3630	4070	30	3	1.55	6660 (4)	6680

**NOTES:**

1. Change in the specimen's length before and after the creep test.
2. Permanent strain over the 1.43-inch central section, as measured with a tool-maker's microscope.
3. Specimens were re-tested at room temperature.
4. Specimen was re-tested at its creep test temperature.

TABLE 5

WITH-GRAIN FLEXURE STRENGTH TESTS ON CYLINDRICAL  
SPECIMENS MACHINED FROM REGULARLY MOLDED P-03 PLATE STOCK

SPECIMEN DENSITY	FLEXURAL STRENGTH		ELECTRICAL <sup>(1)</sup> RESISTIVITY
	3-POINT TEST	4-POINT TEST	
(g/cc)	(psi)	(psi)	( $\times 10^{-3}$ ohm-cm)
A. Plunger pin lot No. 584: Qualification tests			
1.809; 1.821	7855; 8050	6205; 7370	1.990; 1.840
1.823; 1.830	8195; 9455	7760; 8050	1.605* <sup>*</sup> ; 1.570*
1.832; 1.832	10,475; 8290	7470; 7420	1.745; 1.800
1.843; 1.845	9795; 10,380	9070; 8630	1.615; 1.615
1.853; 1.853	10,960; 11,445	9215; 9600	1.530; 1.550
Average and standard deviation:			
1.834 (0.014)	9490 (1320)	8080 (970)	----
B. Plunger pin lot No. 148: Tests on cut-down pins			
1.783; 1.793	5990; 7160	---- (2)	---- (2)
1.811; 1.813	8765; 8290	----	----
1.814; 1.817	8135; 8915	----	----
1.818; 1.820	8420; 8630	----	----
1.821; 1.821	8718; 9394	----	----
1.825; 1.825	8955; 9010; 9100	----	----
1.830; 1.845	9355; 10,090	----	----
Average values:			
1.820	8600	----	----

NOTES:

1. Electrical resistivity was determined before the flexure tests.
2. Not determined.

\* These resistivity values do not conform to those for the other specimens, for unknown reasons.

TABLE 6-A  
FLEXURE TESTS ON SPECIMENS WITH SQUARE CROSS SECTION  
MACHINED FROM ISOSTATICALLY MOLDED P-03 GRAPHITE

<u>DENSITY</u> (g/cc)	<u>ORIENTATION</u>	<u>FLEXURE STRENGTH</u> (psi)	<u>MISCELLANEOUS</u>
A. Flexure bars machined from Sample No. 1			
1.88	RADIAL	6225, 7160 7160, 8030	Regular specimen size,* four tests. Average flexure strength = 7143 psi
1.88	AXIAL	7020, 7075 7290 7500, 7740	Specimen size 1/4" x 1/4" x 2"; spans 1/2 and 1 1/2" five tests. Average flexure strength = 7325 psi, standard deviation = 300 psi.
B. Flexure bars machined from Sample No. 2			
1.839, 1.840	AXIAL	5760, 6460	Regular specimen size,* eight tests. Average flexure strength = 6470 psi, standard deviation = 500 psi.
1.844, 1.845	AXIAL	6450, 6250	
1.848, 1.853	AXIAL	6590, 6080	
1.860, 1.872	AXIAL	6890, 7320	

\*Nominal size 0.43 x 0.43 x 3.50 inches; outer span 3.0 inches, inner span 1.0 inch

TABLE 6-B

FLEXURE TESTS ON SPECIMENS WITH SQUARE CROSS SECTION  
MACHINED FROM REGULARLY MOLDED P-03 GRAPHITE

<u>DENSITY</u> (g/cc)	<u>ORIENTATION</u>	<u>FLEXURAL STRENGTH</u> (psi)	<u>MISCELLANEOUS</u>
A. Sample No. 3, Plate stock			
1.841; 1.849	W. G.	8190, 8240	Regular specimen size*
B. Sample No. 4-a, Cylindrical stock (4.5"D, 4.5"L)			
1.849, 1.851	A. G.	6400, 6500	Regular specimen size*, seven tests. Average flexure strength = 6330 psi Standard deviation = 500 psi
1.854, 1.854	A. G.	6150, 6580, 6930	
1.857, 1.857	A. G.	5330, 6440	
C. Sample No. 4-c, Cylindrical stock (4.5"D, 4.5"L)			
1.849	W. G.	6020, 6170	Regular specimen size*, six tests. Average flexure strength = 6190 psi.
1.851	W. G.	5800, 6060	
1.852	W. G.	6630	
1.853	W. G.	6440	
1.847	A. G.	6100	Regular specimen size*, six tests. Average flexure strength = 6930 psi.
1.850	A. G.	7000, 7190	
1.851	A. G.	7010, 7300	
1.854	A. G.	6970	
D. Sample No. 4-d, Cylindrical stock (4.5"D, 4.5"L)			
1.839	W. G.	5760	Regular specimen size*, six tests. Average flexure strength = 6060 psi
1.845	W. G.	5720	
1.849	W. G.	6600	
1.852	W. G.	5960, 6400	
1.855	W. G.	5920	
1.848	A. G.	6480, 6630, 6850	Regular specimen size*, six tests. Average flexure strength = 6540 psi
1.851	A. G.	6520, 6710	
1.852	A. G.	6030	

\*Nominal size 0.43 x 0.43 x 3.50 inches, inner span 1.0 inch, outer span 3.0 inches

TABLE 7-A
COMPRESSION TEST SUMMARY FOR ISOSTATICALLY MOLDED P-03 GRAPHITE

<u>SPECIMEN DENSITY</u> (g/cc)	<u>ORIENTATION</u>	<u>COMPRESSIVE STRENGTH</u> (psi)	<u>STRAIN RATE</u>	<u>MISCELLANEOUS</u>
<b>A. Sample No. 1</b>				
1.875	RADIAL	21,410, 21,640, 22,000	0.02	Av. = 21,680 (3 tests) Av. = 22,360 (7 tests) Std. dev. = 625
1.880	AXIAL	21,380, 21,960, 22,060	0.02	
1.880	AXIAL	22,110, 22,730, 22,930	0.02	
1.880	AXIAL	23,320	0.02	
<b>B. Sample No. 2</b>				
1.824	AXIAL	21,275	0.02	Av. = 20,920 (10 tests)  Av. = 20,925 (5 tests) Std. dev. = 1225 (All tests on Sample 2)
1.837, 1.838	AXIAL	20,050, 20,150	0.02	
1.843, 1.845, 1.846	AXIAL	18,150, 20,500, 21,350	0.02	
1.852	AXIAL	21,300	0.02	
1.861, 1.863, 1.865	AXIAL	23,475, 23,825, 22,100	0.02	
1.836, 1.839	AXIAL	20,650, 19,875	0.20	
1.846, 1.849	AXIAL	20,700, 21,000	0.20	
1.857	AXIAL	22,400	0.20	

TABLE 7-B  
COMPRESSION TEST SUMMARY FOR REGULARLY MOLDED P-03 GRAPHITE

<u>SPECIMEN DENSITY</u> (g/cc)	<u>ORIENTATION</u>	<u>COMPRESSIVE STRENGTH</u> (psi)	<u>MISCELLANEOUS</u>
A. Sample No. 3, Plate stock			
1.834, 1.837	W. G.	21,400, 22,400	Average density = 1.843 g/cc Average C.S. = 22,180 psi Std. deviation = 560 psi
1.843, 1.844	W. G.	22,900, 22,300	
1.853	W. G.	21,900	
B. Sample No. 4-a, Cylindrical stock (4.5"D, 4.5"L)			
1.828	A. G.	18,100	Average density = 1.845 g/cc Average C.S. = 18,320 psi Std. deviation = 1090 psi No. of tests = 12
1.842	A. G.	16,600	
1.842, 1.843	A. G.	17,400, 17,750	
1.847, 1.849	A. G.	18,600, 17,900	
1.850	A. G.	18,200, 20,600	
1.851, 1.852	A. G.	17,800, 19,800	
1.853, 1.854	A. G.	17,900, 19,200	
C. Sample No. 4-c, <sup>(1)</sup> Cylindrical stock (4.5"D, 4.5"L)			
1.844, 1.846	A. G.	18,600, 21,100	Average density = 1.850 Average C.S. = 20,320 psi Std. deviation = 817 No. of tests = 12
1.847	A. G.	19,800, 20,800	
1.849, 1.850	A. G.	19,400, 20,300*	
1.851	A. G.	21,000, 21,200	
1.852	A. G.	20,000, 20,100	
1.853	A. G.	20,400, 21,100	
D. Sample No. 4-d, <sup>(2)</sup> Cylindrical stock (4.5"D, 4.5"L)			
1.833	A. G.	17,500	Average density = 1.846 g/cc Average C.S. = 19,530 psi Std. deviation = 1,478 psi No. of tests = 12
1.840	A. G.	14,400, 19,700	
1.843	A. G.	21,800	
1.844	A. G.	18,800, 20,200	
1.846, 1.848	A. G.	19,500, 20,000	
1.850	A. G.	19,200	
1.855	A. G.	19,600, 19,900	
1.864	A. G.	20,700	

<sup>^</sup> Strain gage tests

(1) Serial number 22850, radiographically rejected for low-density regions.

(2) Serial number 22854, radiographically acceptable structure.

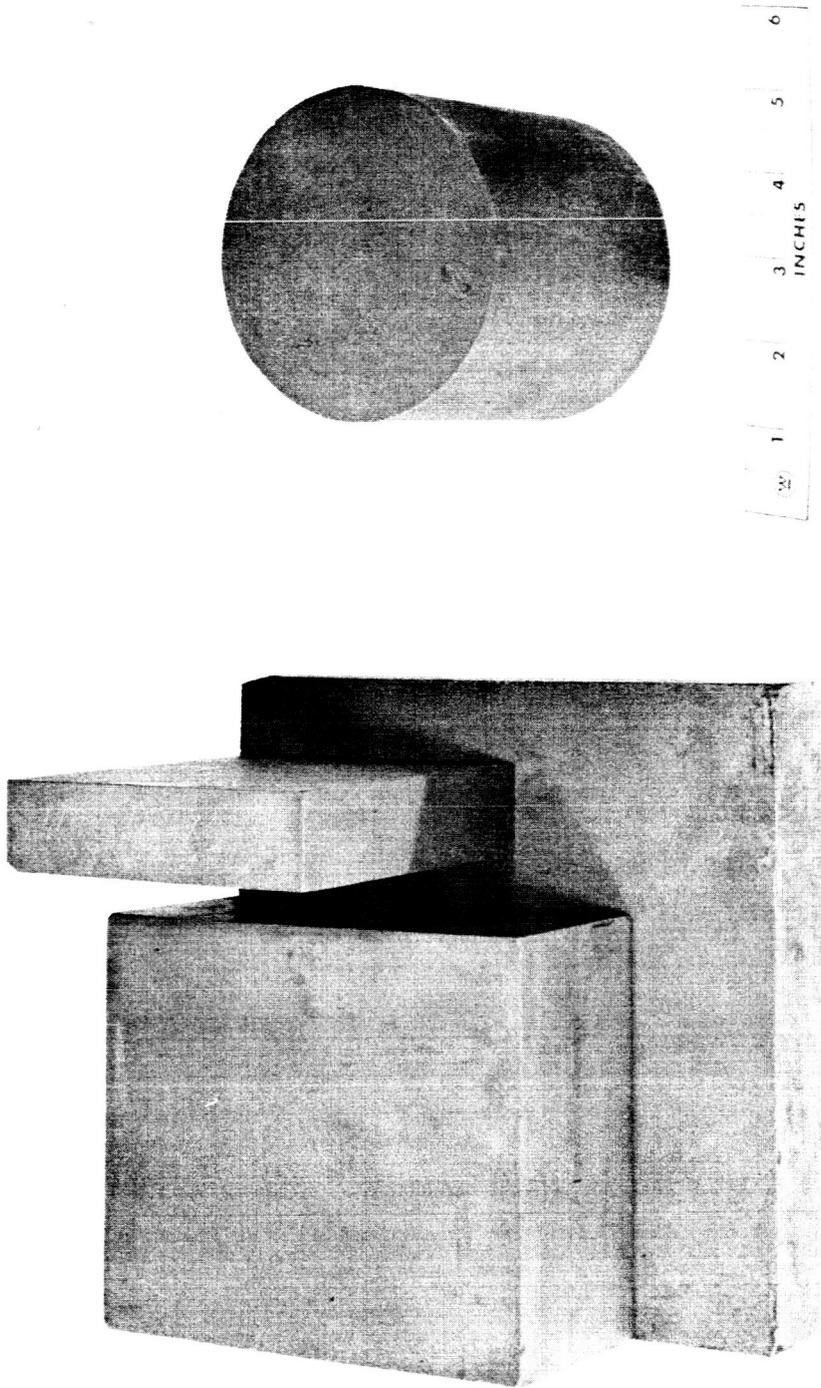


FIGURE 1-a

TYPICAL APPEARANCE OF REGULARLY MOLDED P-03 STOCK. ALL DISCOLORATIONS RESULTED FROM ROUTINE HANDLING.

LEFT: ASSORTED SAMPLES OF P-03 STOCK, SHOWING THE ORIGINAL MOLDED SURFACES.

RIGHT: TYPICAL ROUGH-MACHINED CYLINDER PREPARED FROM THE 4.5" D x 4.5" L STOCK.

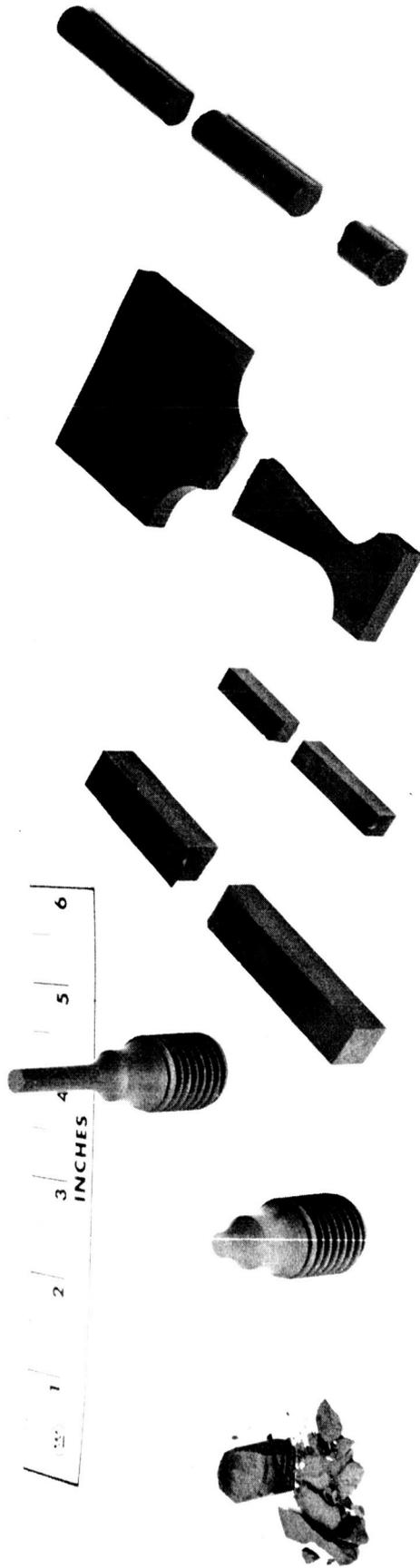


FIGURE 1-b

SPECIMENS USED FOR MECHANICAL EVALUATION TESTS ON P-03 GRAPHITE.  
FROM LEFT TO RIGHT, THESE TYPICAL BROKEN SPECIMENS ARE IDENTIFIED AS FOLLOWS:

- COMPRESSION CYLINDER;
- L-2 TENSILE SPECIMEN (SINGLE FRACTURE);
- REGULAR FLEXURE BAR (SQUARE CROSS-SECTION);
- SMALL FLEXURE BAR (SQUARE CROSS-SECTION);
- VIBRATION SPECIMEN;
- ROUND FLEXURE BAR (BROKEN IN BOTH THE THREE-POINT AND FOUR-POINT LOADING).

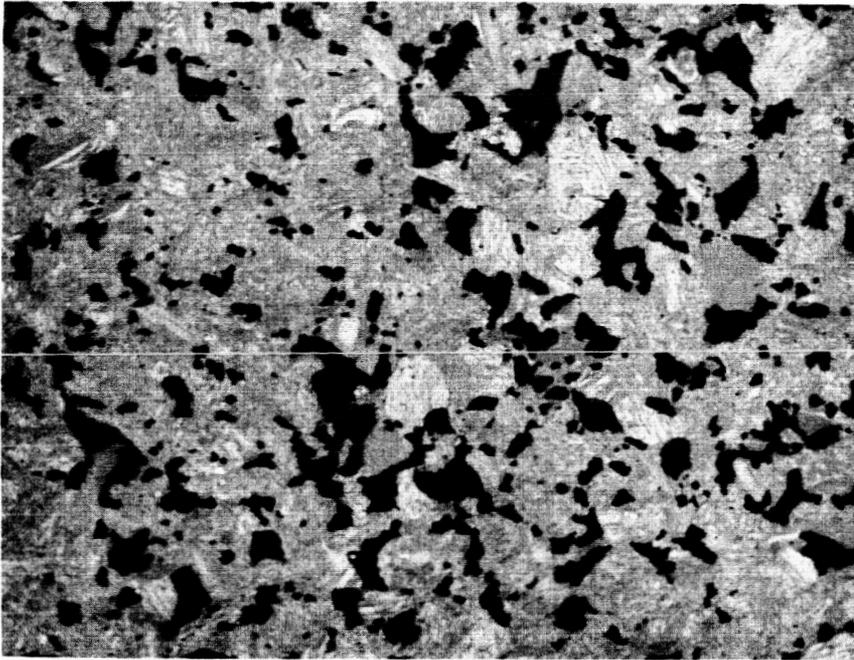


FIGURE 2-b: ATJ MICROSTRUCTURE

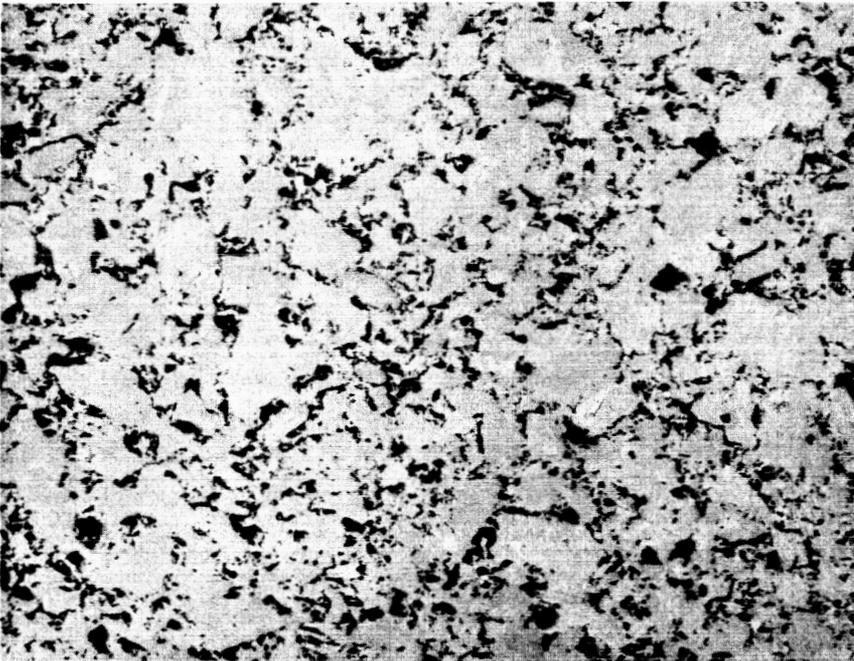


FIGURE 2-a: P-03 MICROSTRUCTURE

100x

FIGURE 2

COMPARISON OF MICROSTRUCTURE BETWEEN THE REGULARLY  
MOLDED P-03 AND ATJ GRAPHITES.

SCALE IN MILLIMETERS  
0 1/2 1

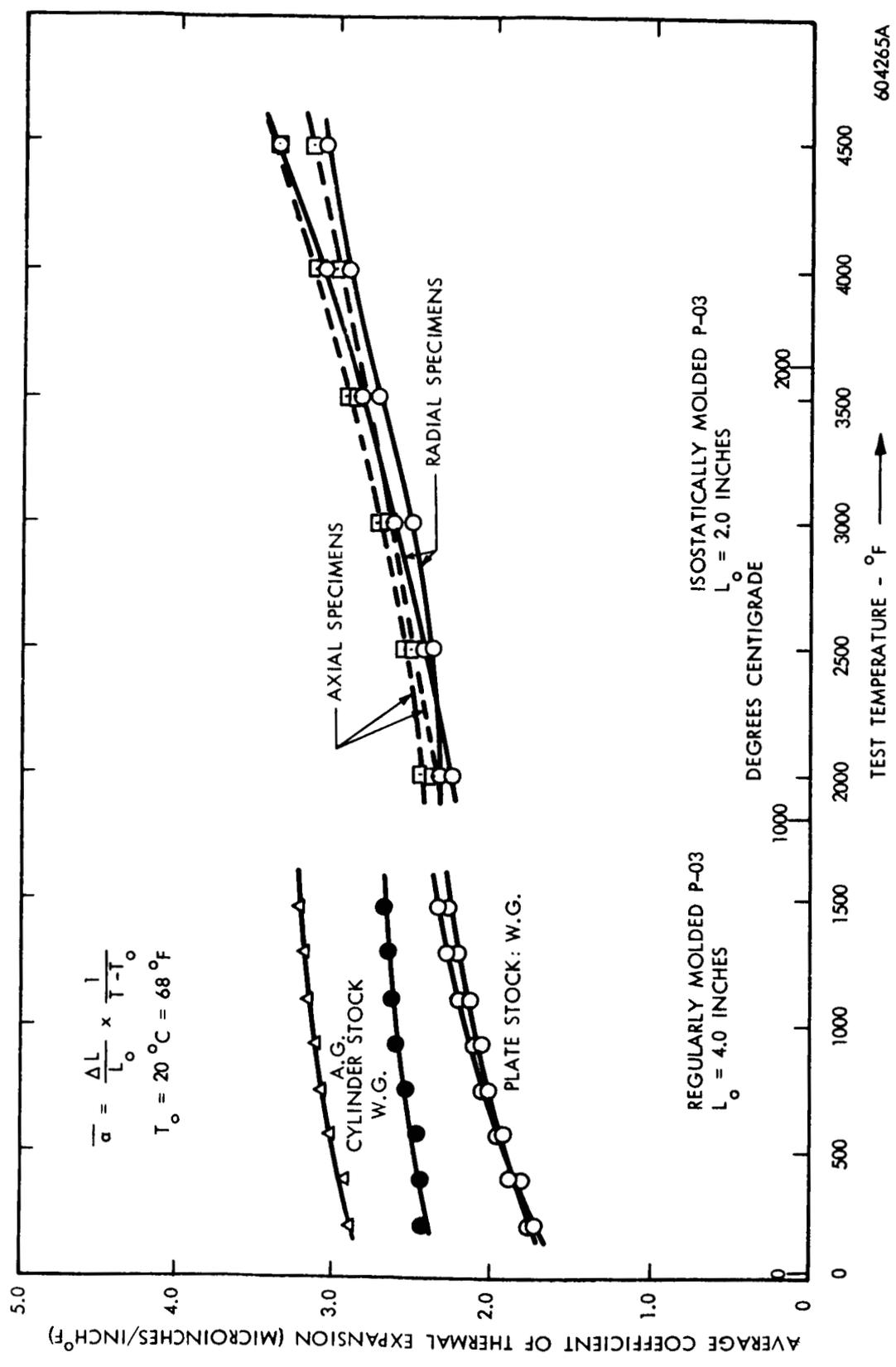


FIGURE 3 - THERMAL EXPANSION OF P-03 GRAPHITE AS A FUNCTION OF TEMPERATURE. TEST RESULTS FOR THE REGULARLY MOLDED P-03 ARE AVAILABLE ONLY TO 1500 °F, WHILE THE ISOSTATICALLY MOLDED P-03 WAS NOT STUDIED BELOW 2000 °F.

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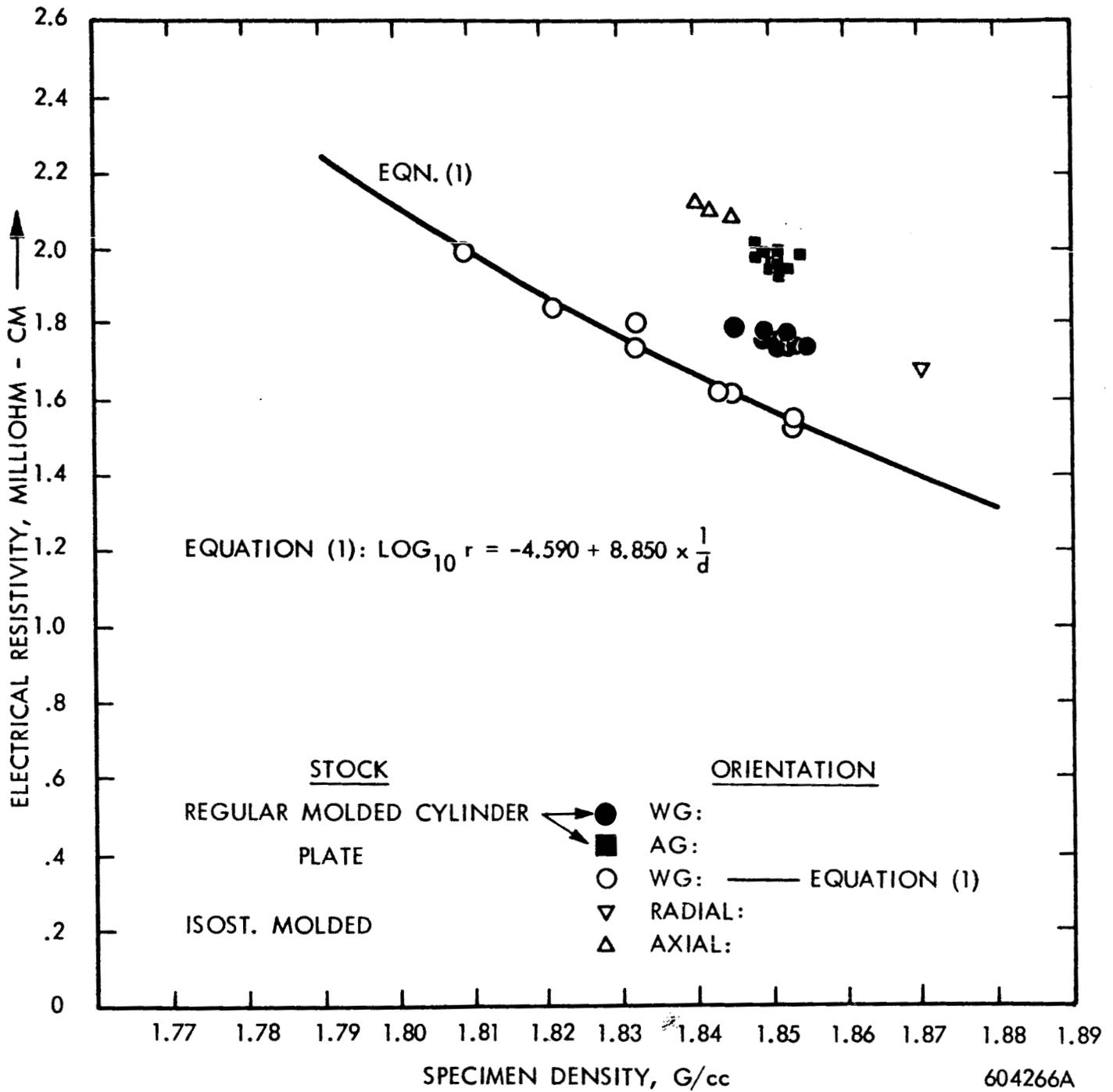


FIGURE 4 ELECTRICAL RESISTIVITY OF P-03 GRAPHITE AT ROOM TEMPERATURE, AS A FUNCTION OF DENSITY. EQUATION NO. 1 GIVES THE RELATIONSHIP FOR WITH-GRAIN SPECIMENS FROM REGULARLY MOLDED PLATE STOCK.

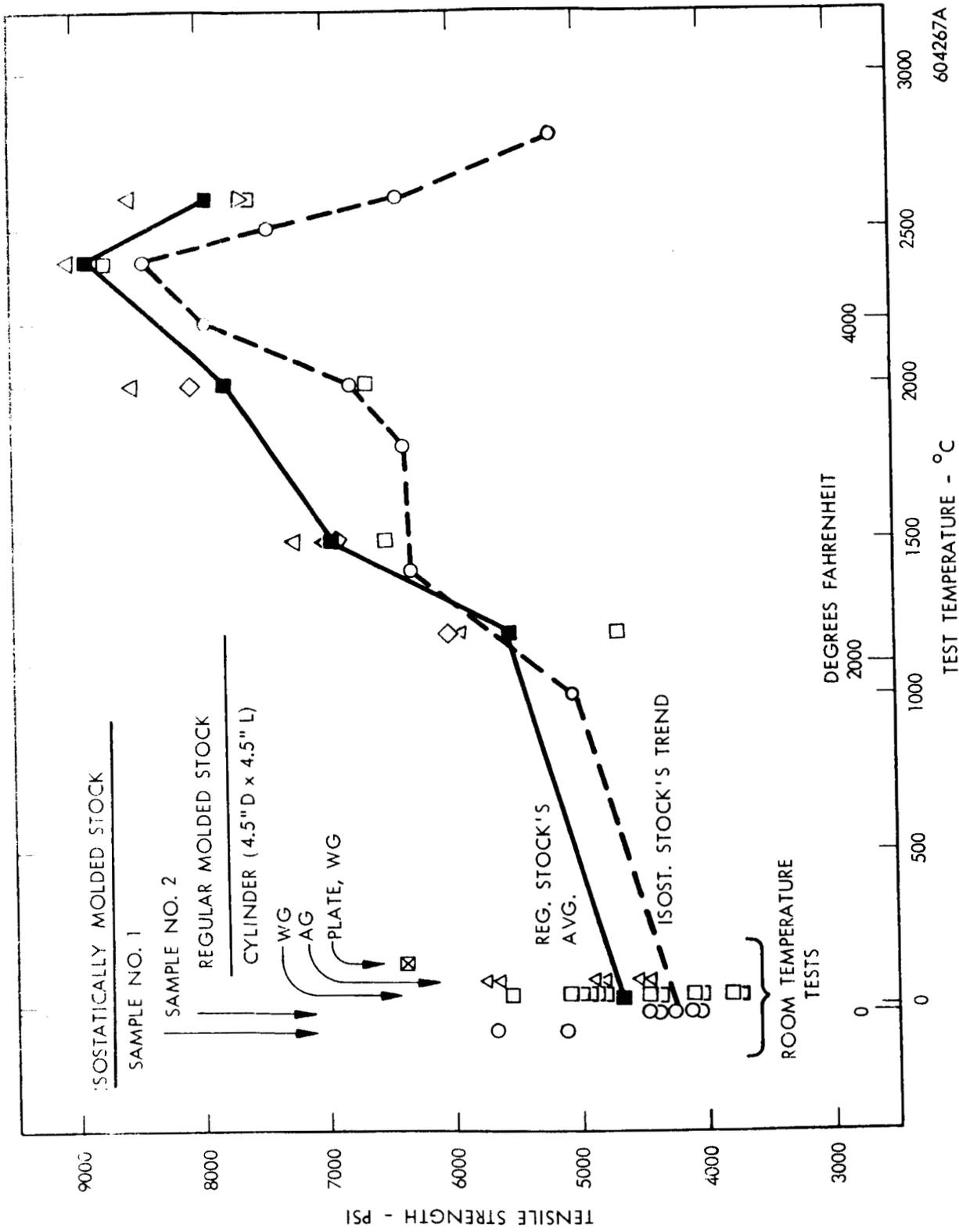


FIGURE 5 SUMMARY OF ALL TENSILE STRENGTH TESTS ON PO-3 GRAPHITE, GIVEN AS A FUNCTION OF TEMP.

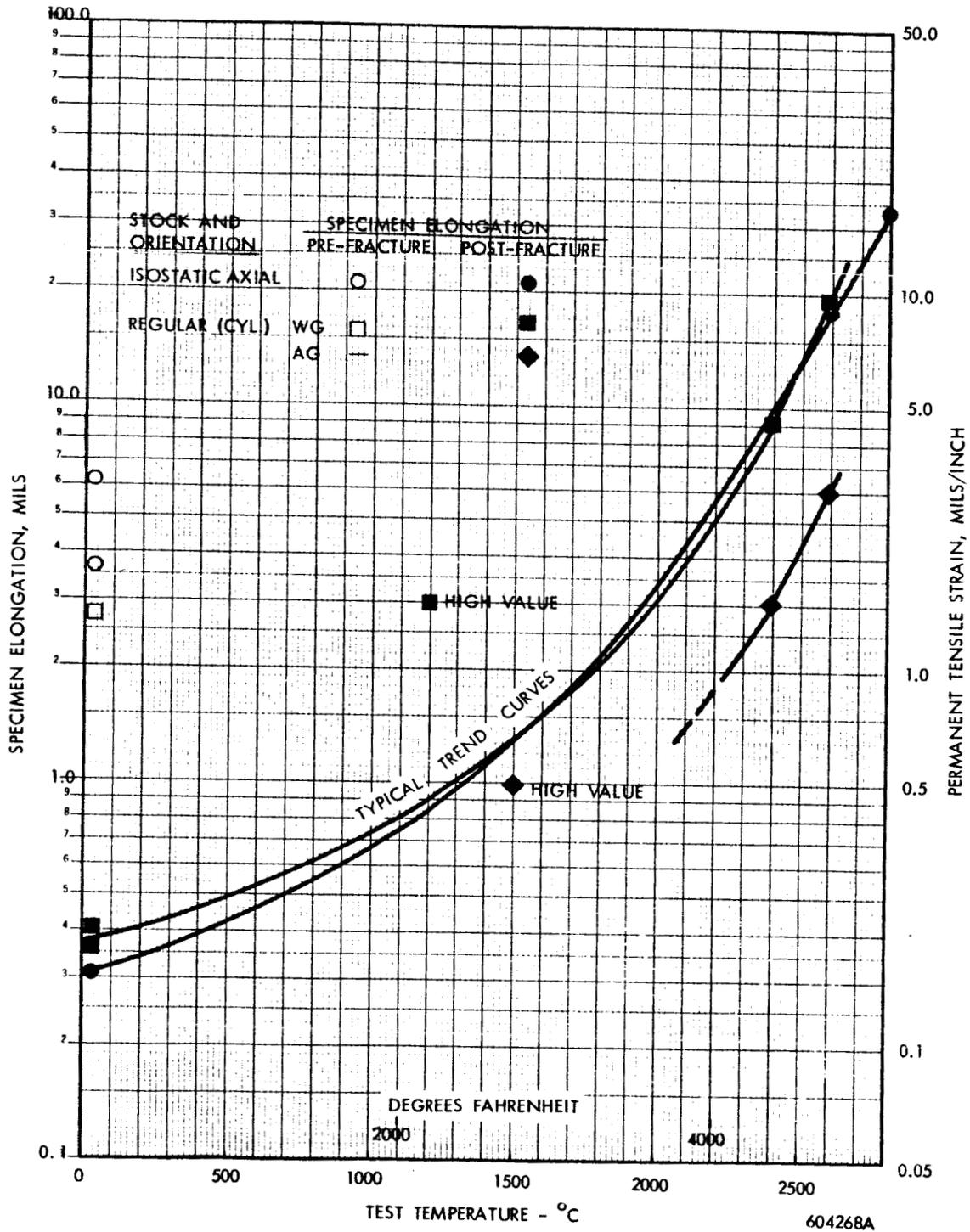


FIGURE 6 PERMANENT TENSILE STRAIN AS A FUNCTION OF TEST TEMPERATURE. ALL SPECIMENS WERE STRAINED AT 0.02 INCH PER MINUTE. THE RELATION BETWEEN THE TOTAL AND PERMANENT STRAINS IS EVIDENT FROM THE ROOM-TEMPERATURE DATA.

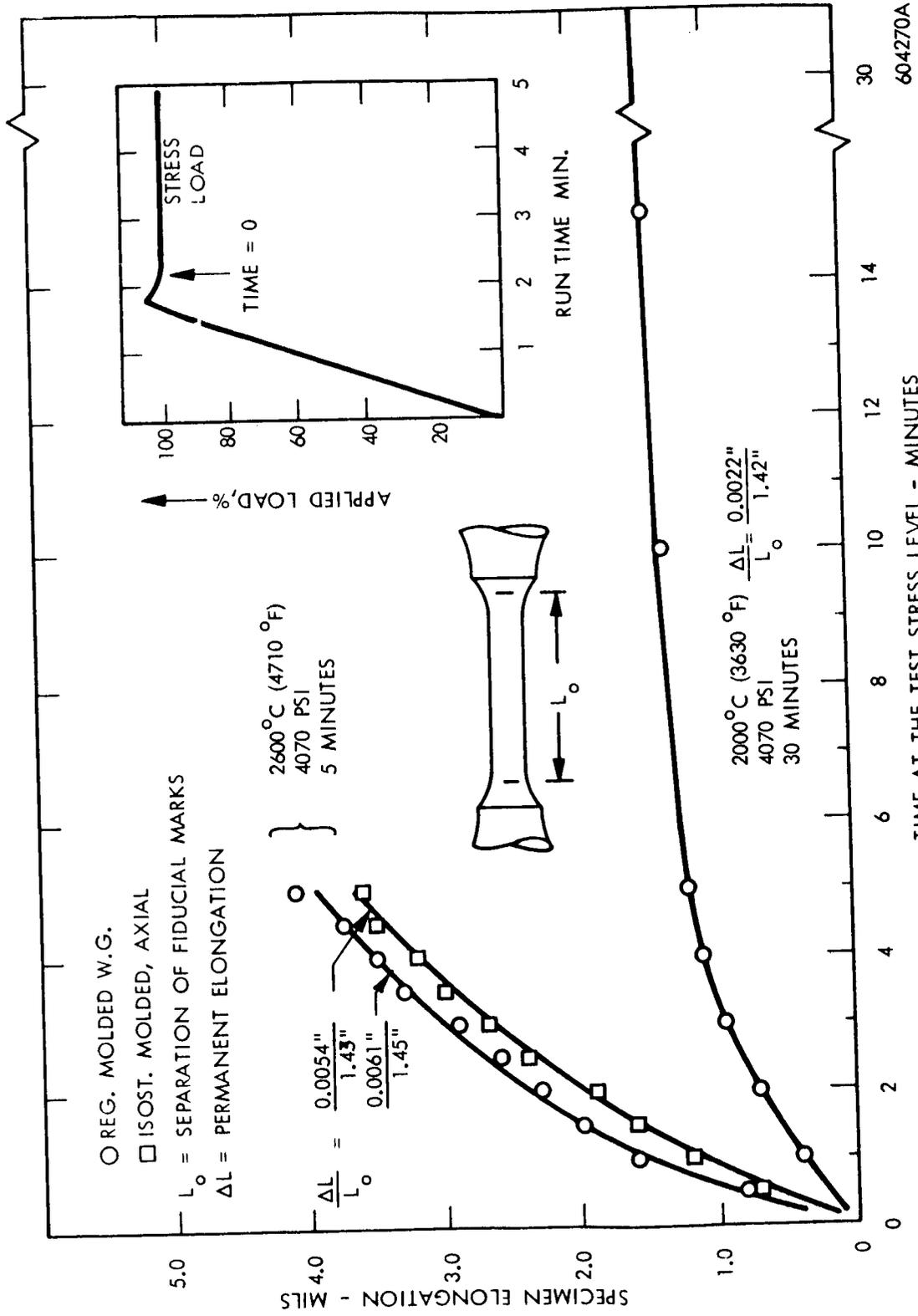


FIGURE 7 PRELIMINARY CREEP CURVES FOR P-03 GRAPHITE AT HIGH TEMPERATURES. THE INSERT SHOWS APPROXIMATE LOADING CURVE, DEMONSTRATING THE SLIGHT INITIAL OVERLOAD APPLIED TO OVERCOME THE FRICTION SLIPPAGE EFFECTS. ELONGATION IN THE GAGE SECTION IS ABOUT 55% OF THE TOTAL ELONGATION.

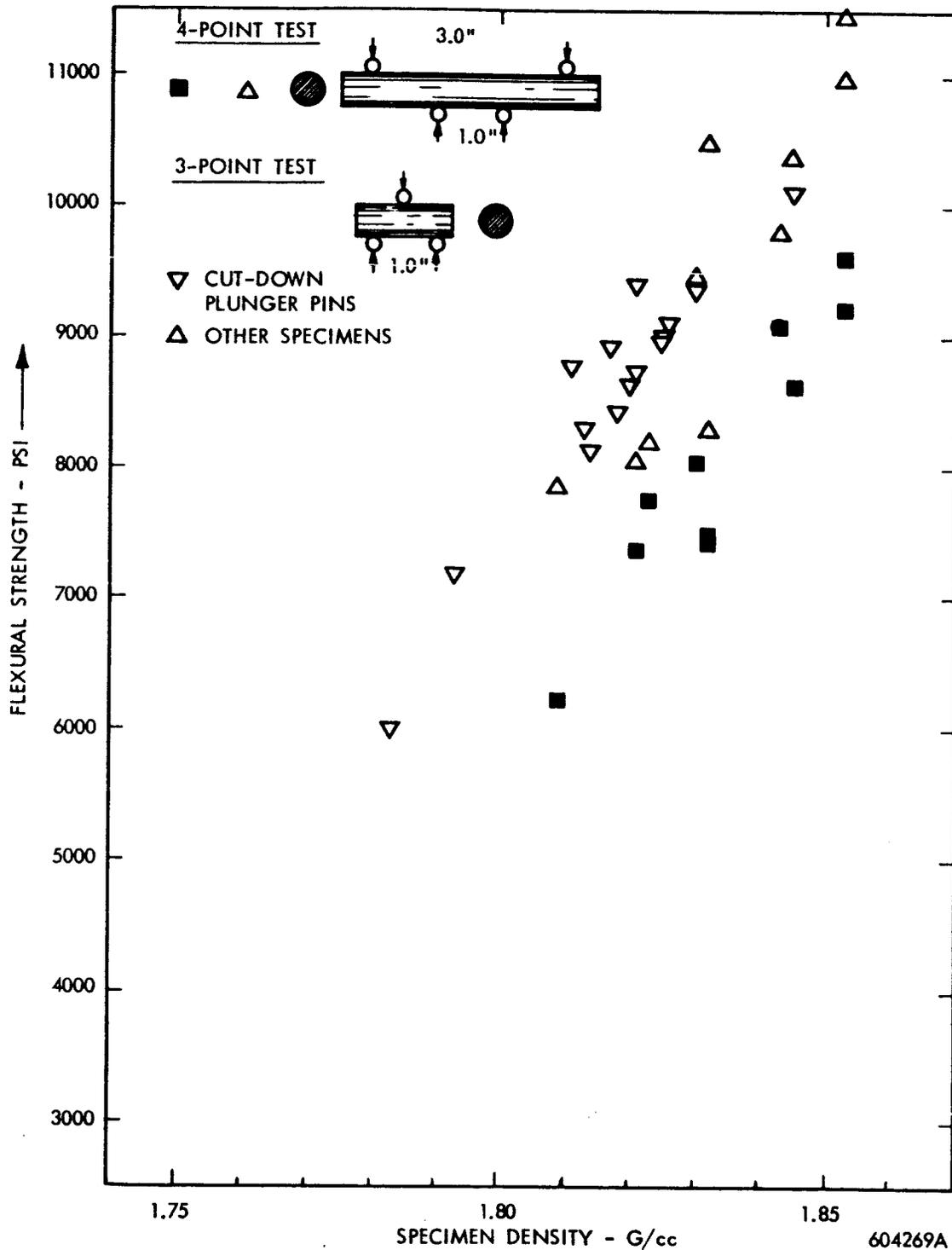


FIGURE 8 SUMMARY OF FLEXURE TESTS ON P-03 SPECIMENS WITH ROUND CROSS-SECTION, AS A FUNCTION OF DENSITY. ALL TESTS WERE MADE ON THE WITH-GRAIN SPECIMENS MACHINED FROM REGULARLY MOLDED PLATE STOCK.

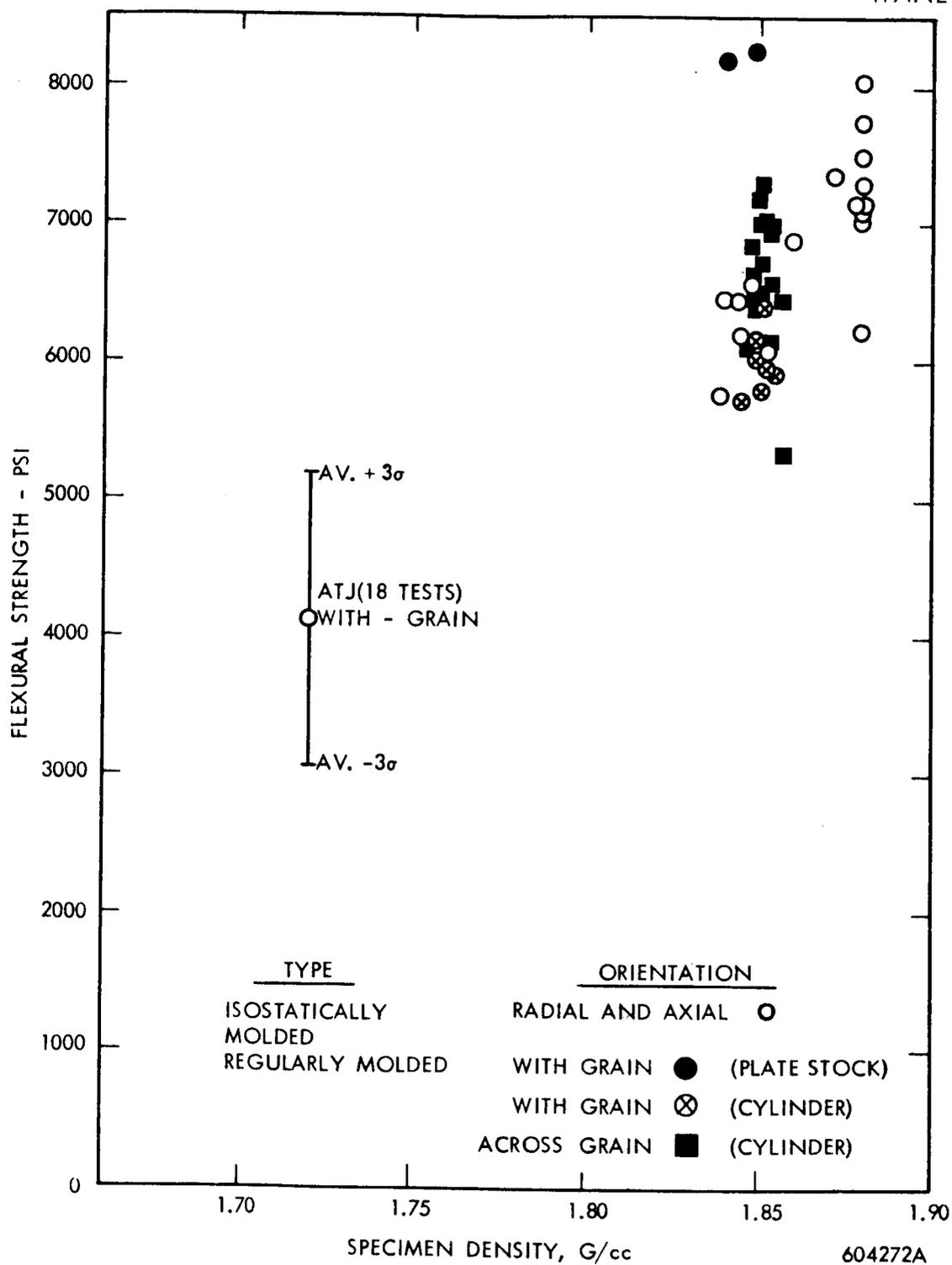


FIGURE 9 SUMMARY OF FLEXURE TESTS ON P-03 SPECIMENS WITH SQUARE CROSS-SECTION, PLOTTED AS A FUNCTION OF DENSITY. WITH-GRAIN TEST RESULTS OF HARDWARE - GRADE ATJ ARE PRESENTED FOR COMPARISON.

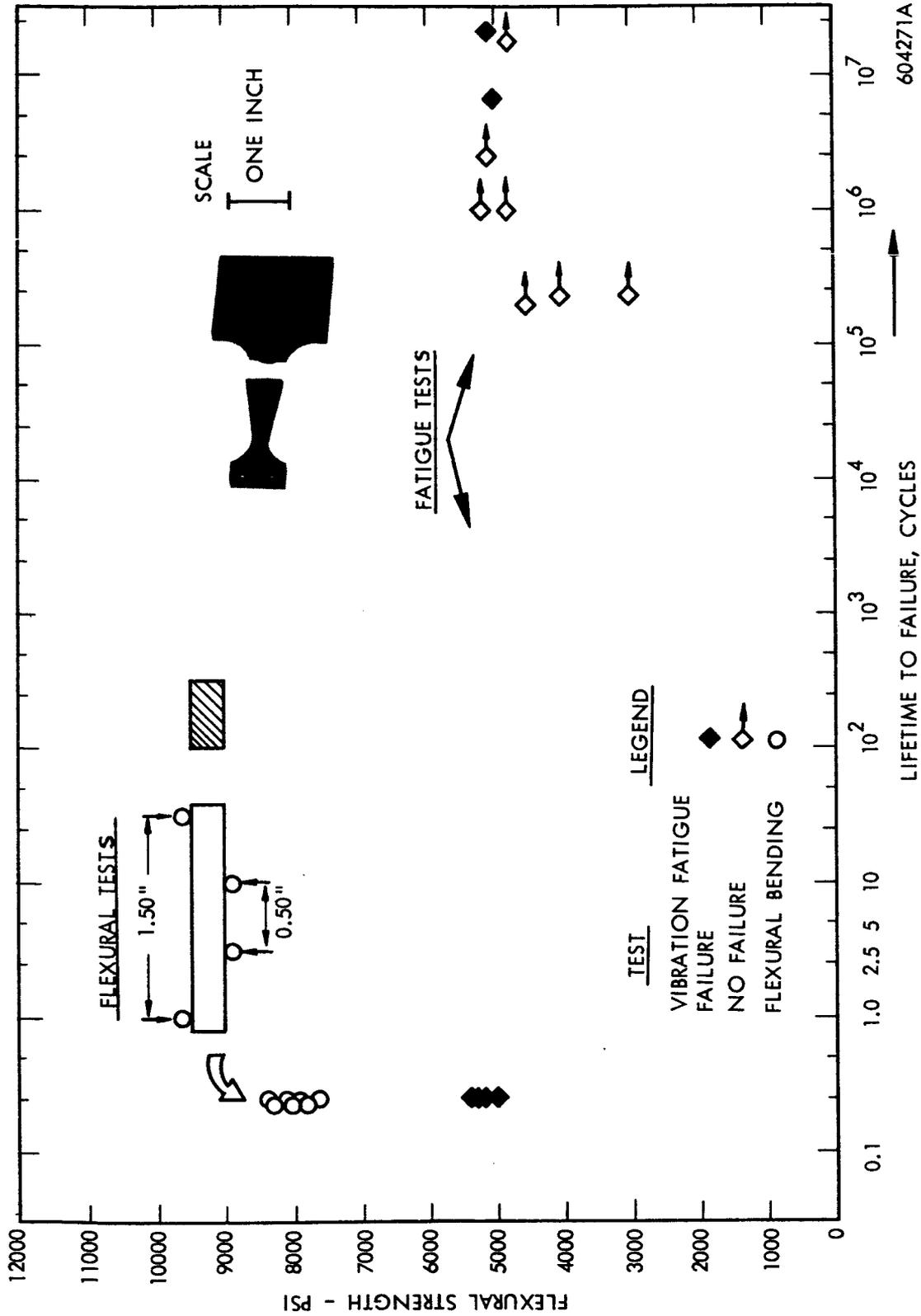
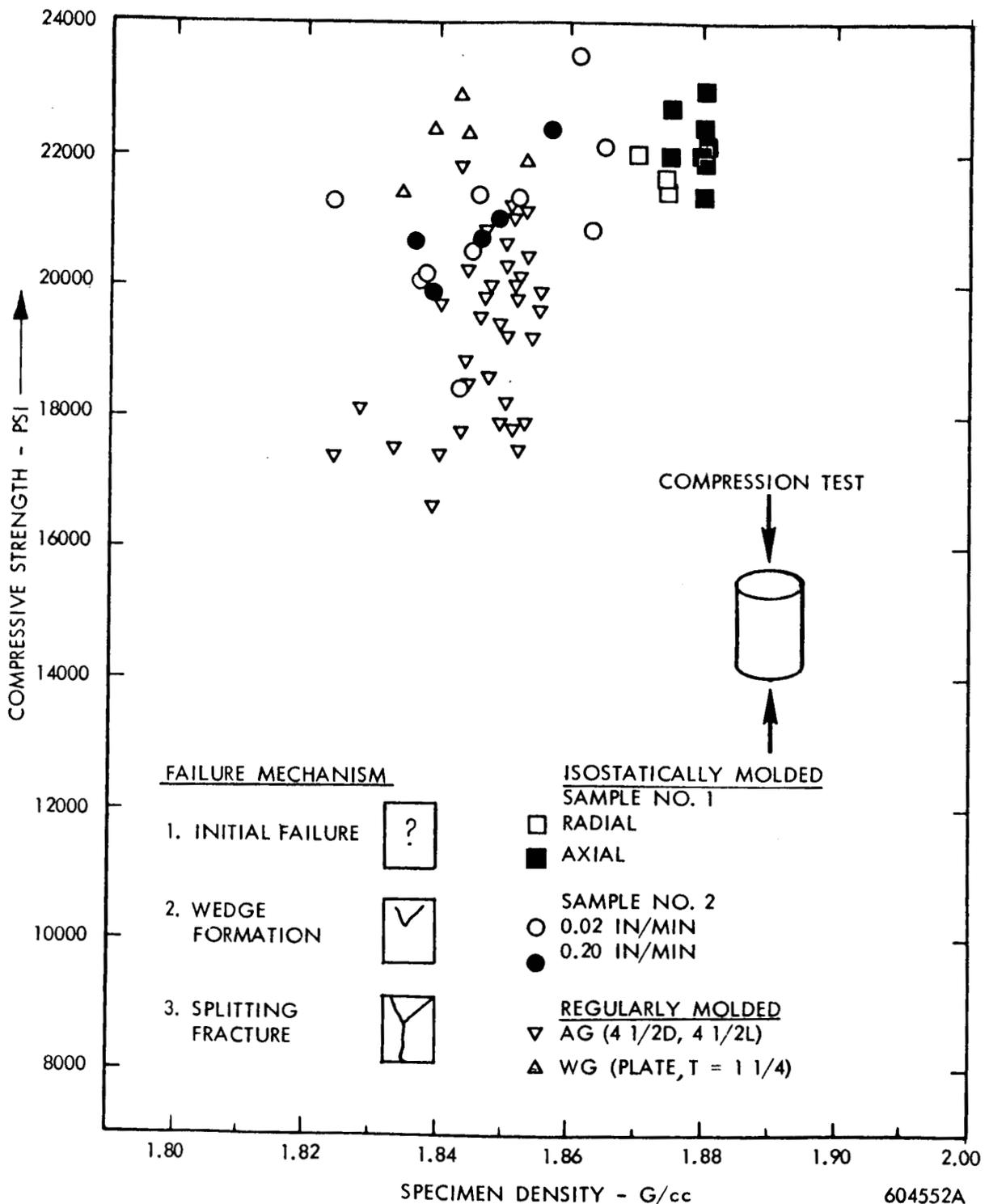


FIGURE 10 BEHAVIOR OF P-03 GRAPHITE IN VIBRATION FATIGUE, TOGETHER WITH COMPARATIVE FLEXURAL BENDING DATA. ALL SPECIMENS WERE MACHINED FROM REGULARLY MOLDED PLATE STOCK IN THE WITH- GRAIN ORIENTATION.



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FIGURE 11 STRENGTH OF P-03 IN COMPRESSION (DETERMINED ON 0.50" D x 0.90"L CYLINDRICAL SPECIMENS), AS A FUNCTION OF DENSITY.

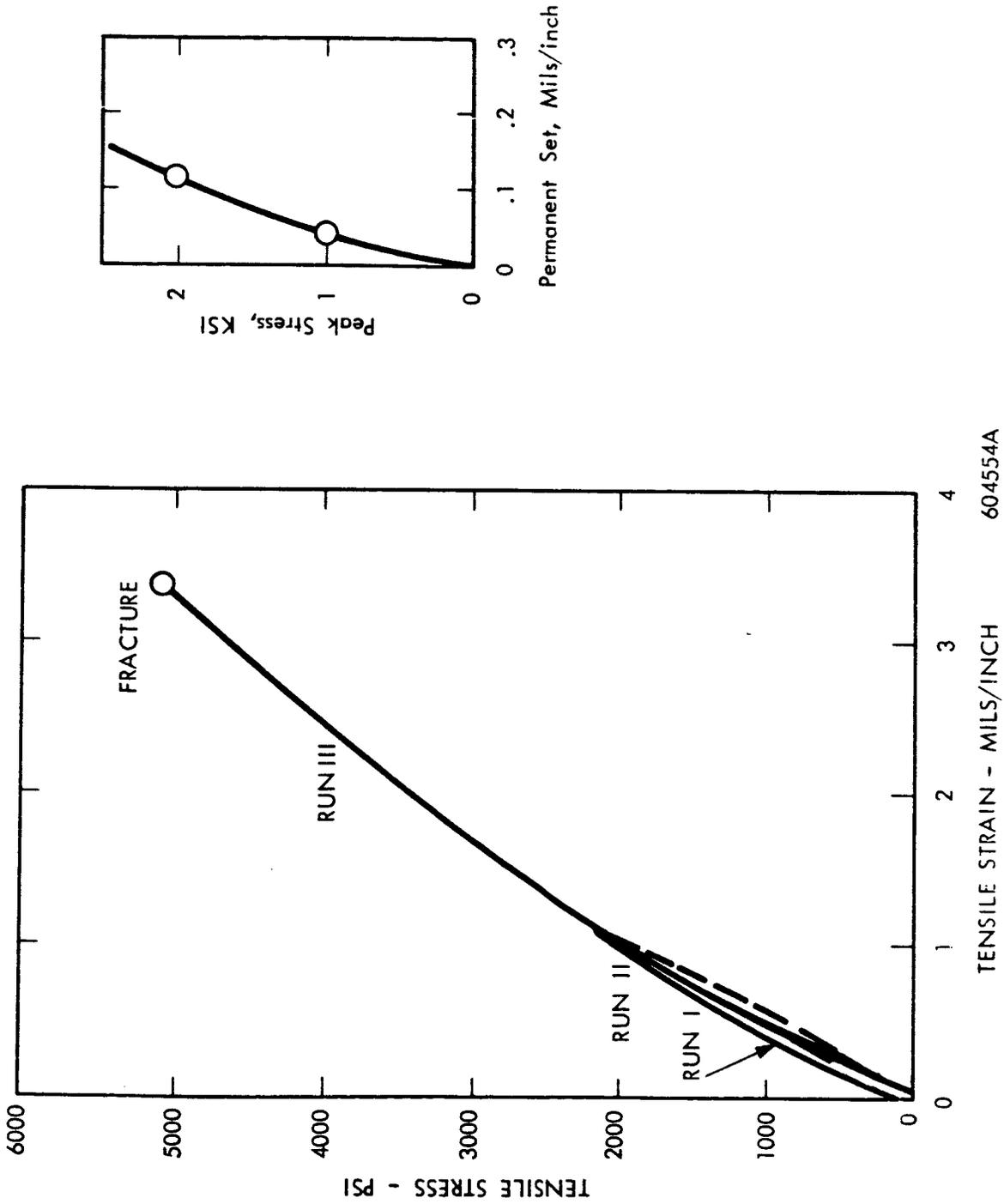


FIGURE 12 STRESS VERSUS STRAIN CURVE FOR ISOSTATICALLY MOLDED P-03, DETERMINED ON A RADIAL SPECIMEN (DENSITY 1.88 g/cc) MACHINED FROM SAMPLE No. 1. THE INSERT SHOWS A MAGNITUDE OF PERMANENT DEFORMATION AS A FUNCTION OF PEAK PRELOAD STRESS.

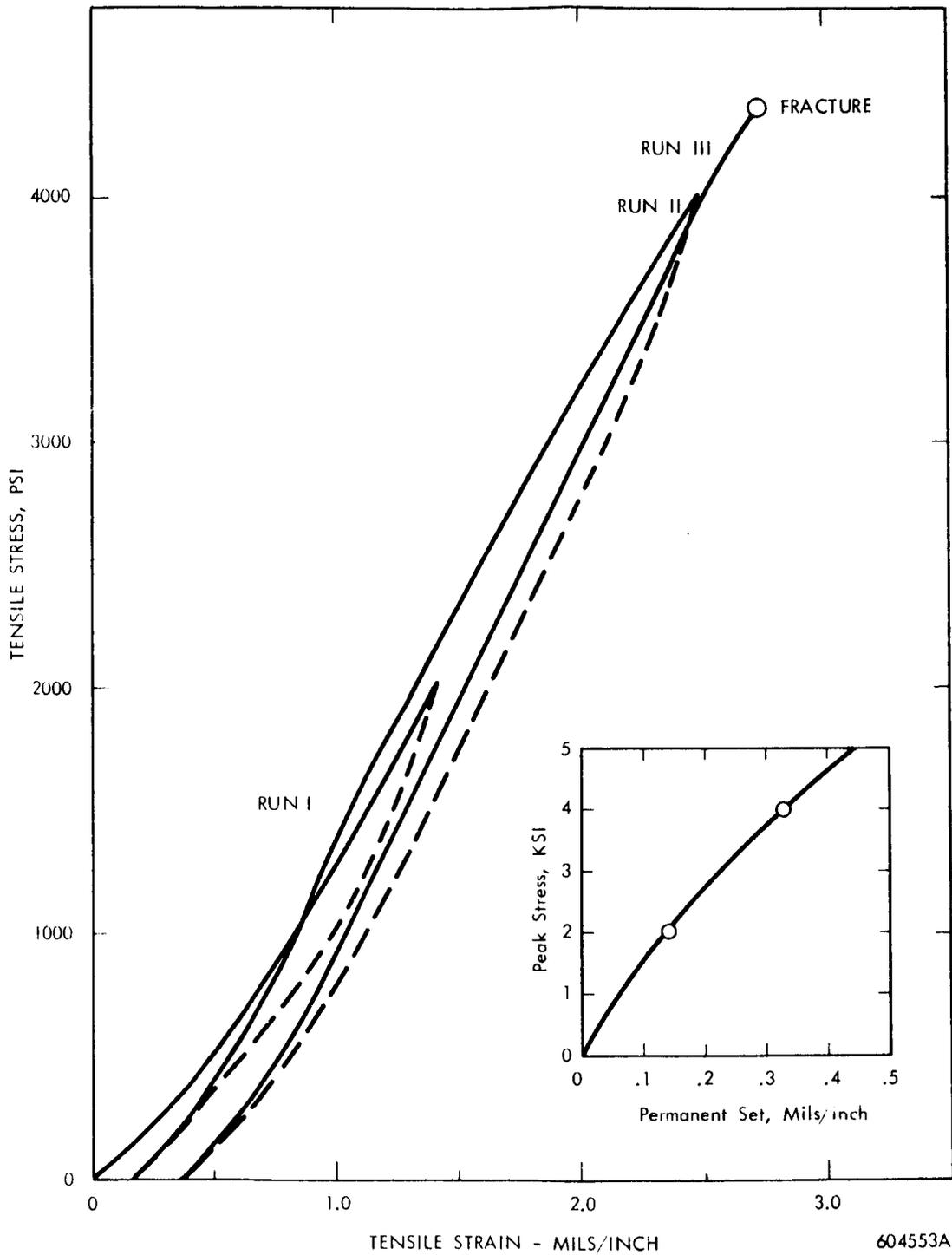


FIGURE 13 TENSILE STRESS VERSUS STRAIN CURVE FOR REGULARLY MOLDED P-03 (SAMPLE NO 4-d: 4.5" D x 4.5" L, CYLINDRICAL STOCK), DETERMINED ON WITH-GRAIN SPECIMEN. BELOW 1000-PSI STRESS LEVEL THE CURVES ARE SUBJECT TO A SLIGHT SYSTEMATIC ERROR CAUSED BY OFF-CENTERED LOADING.

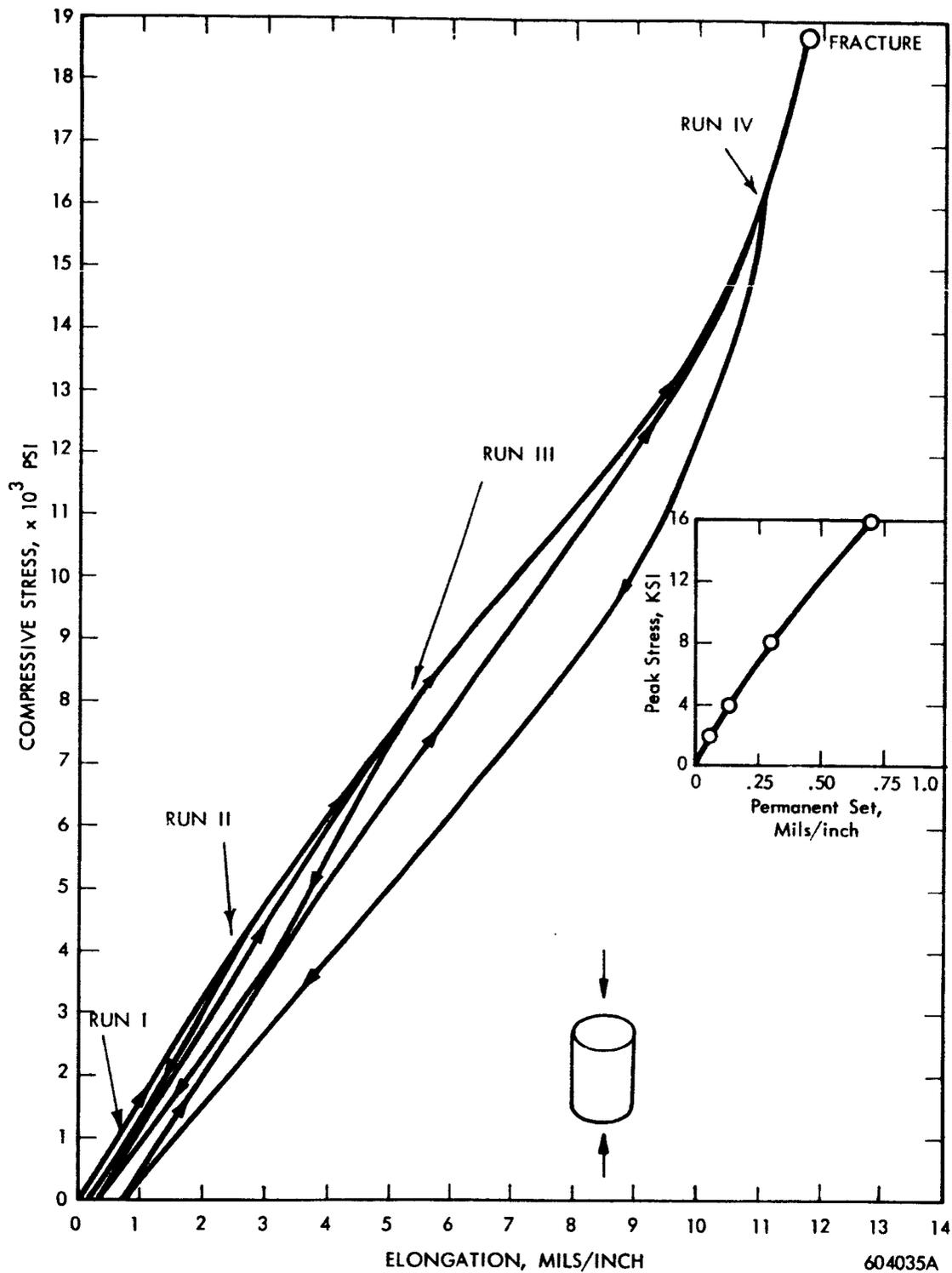


FIGURE 14 COMPRESSIVE STRESS- STRAIN CURVE FOR REGULARLY MOLDED P-03 GRAPHITE (SAMPLE NO. 4-d; 4.5" D x 4.5" L CYLINDRICAL STOCK), AS DETERMINED ON ACROSS-GRAIN SPECIMEN OF 1.844 g/cc DENSITY.

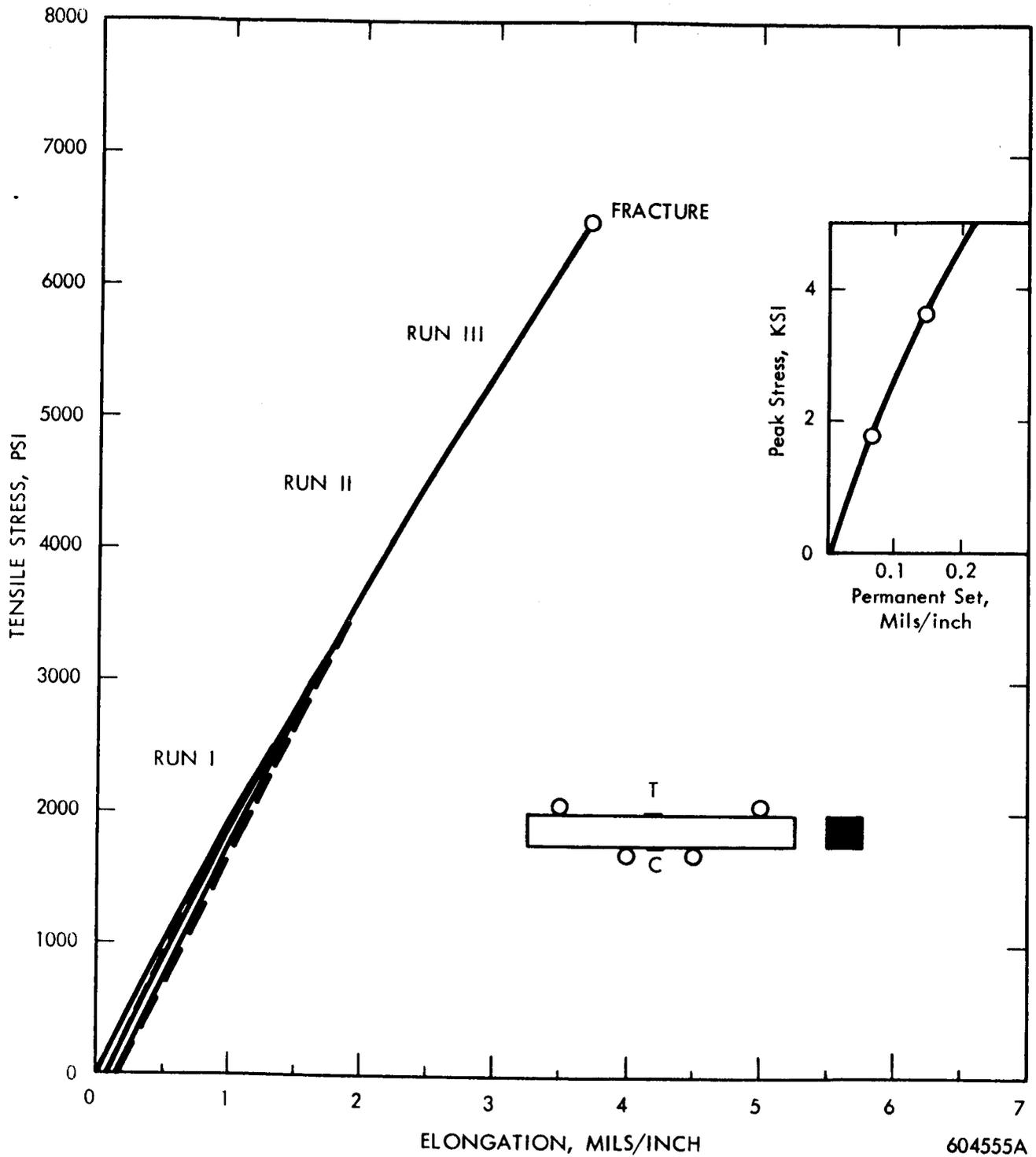


FIGURE 15-a STRESS VERSUS STRAIN CURVE ON THE TENSILE FACE OF A FLEXURE SPECIMEN MACHINED FROM SAMPLE NO.2 OF ISOSTATICALLY MOLDED P-03 (DENSITY 1.840 g/cc) IN THE AXIAL DIRECTION.

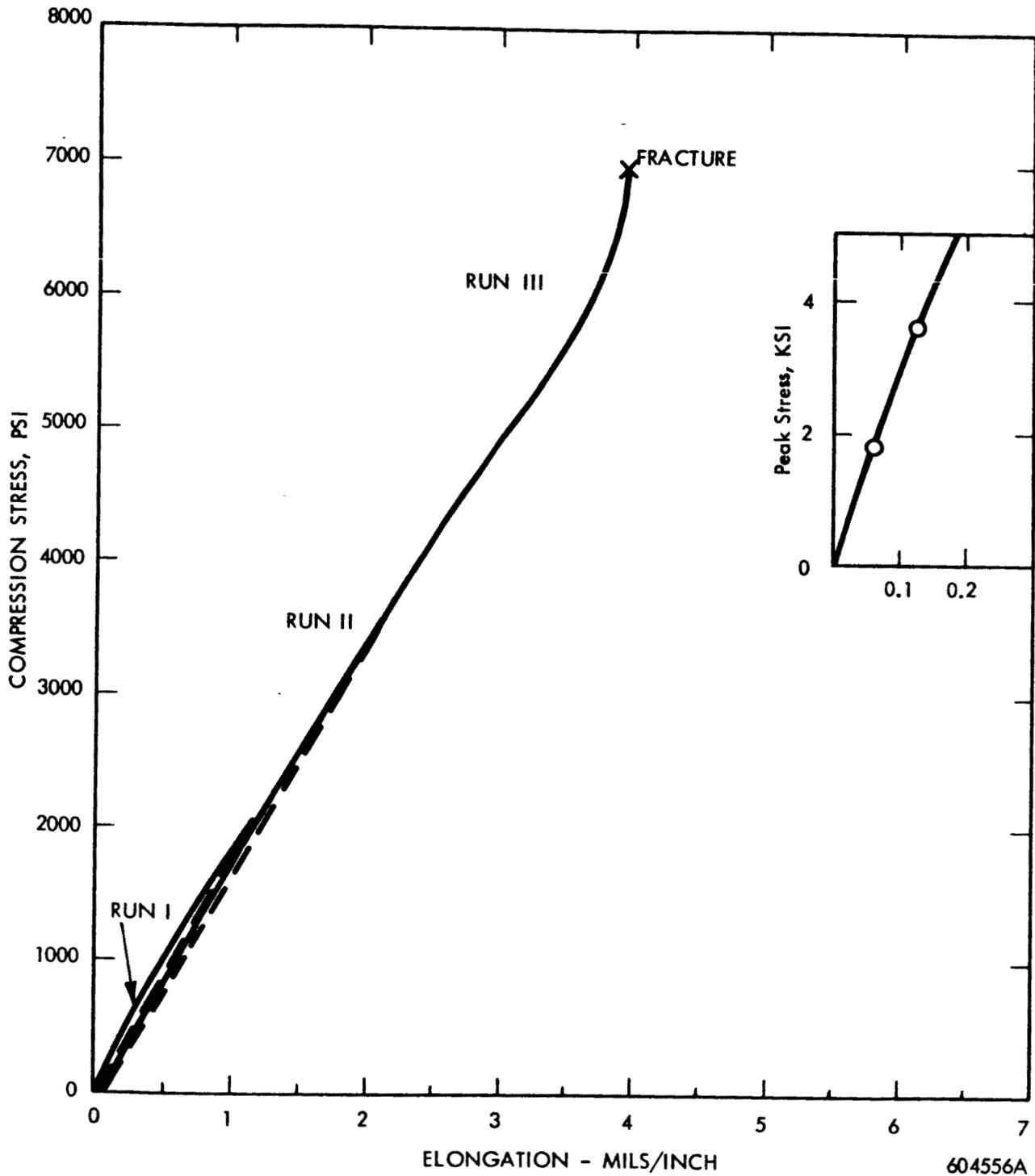


FIGURE 15-b STRESS VERSUS STRAIN CURVE ON THE COMPRESSIVE FACE OF THE SPECIMEN IN FIGURE 15-a.

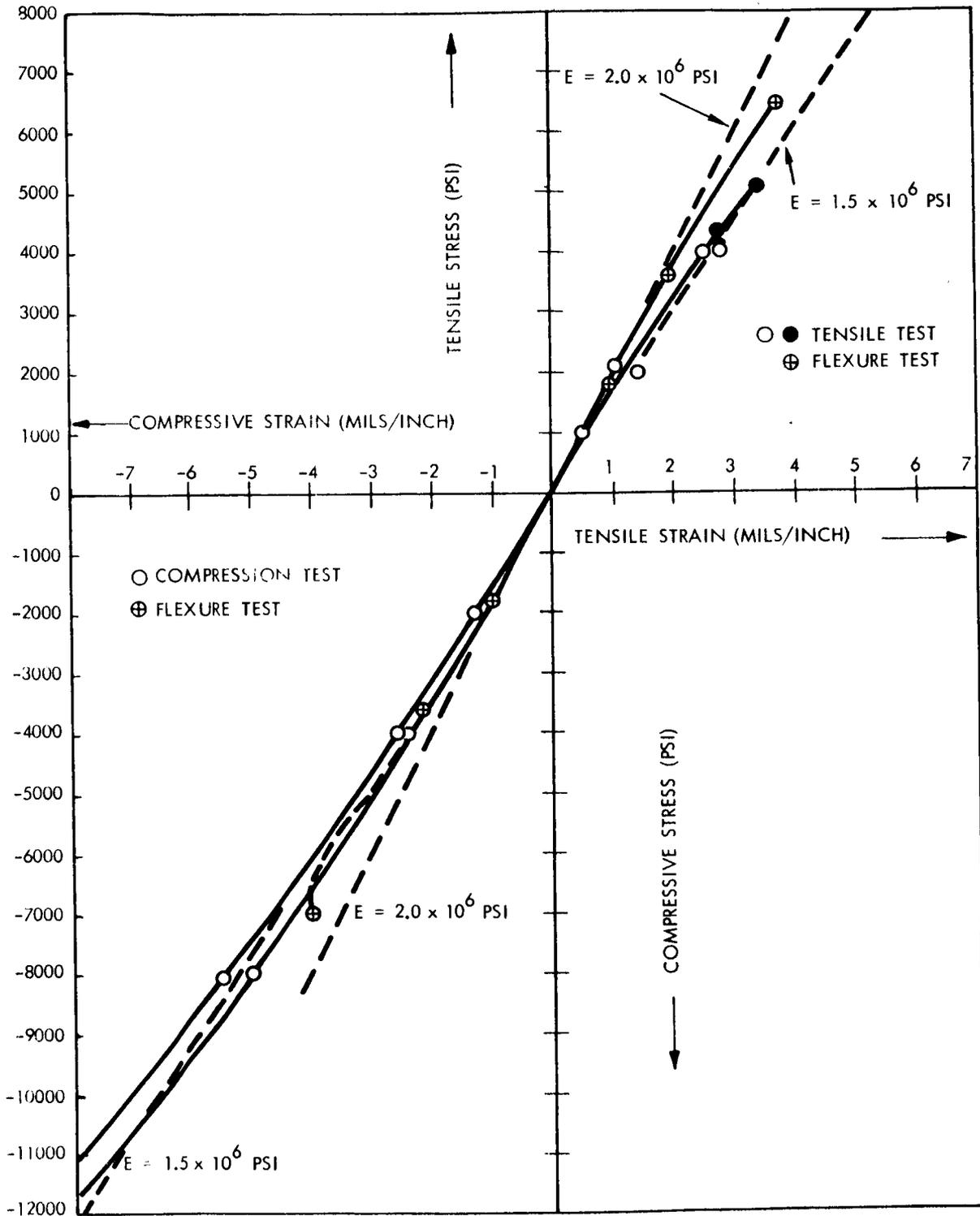


FIGURE 16 GENERALIZED STRESS-STRAIN BEHAVIOR OF P-03 GRAPHITE. NOTE THAT THE SECANT YOUNG'S MODULUS VARIES IN THE  $1.5 - 2.0 \times 10^6$  PSI RANGE.

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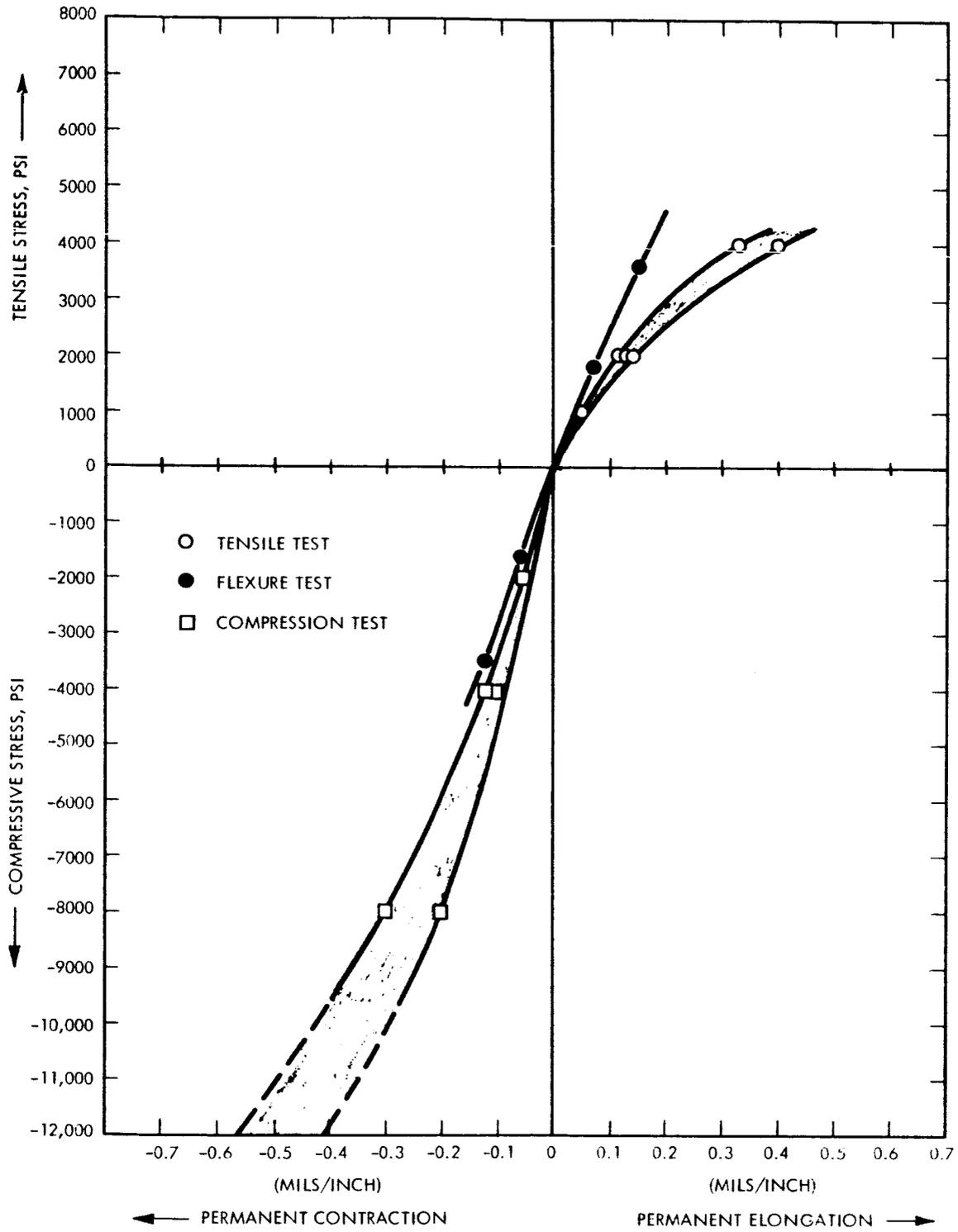


FIGURE 17 PERMANENT SET IN P-03 GRAPHITE  
 AS A FUNCTION OF THE PEAK STRESS APPLIED  
 ON VARIOUS SPECIMENS

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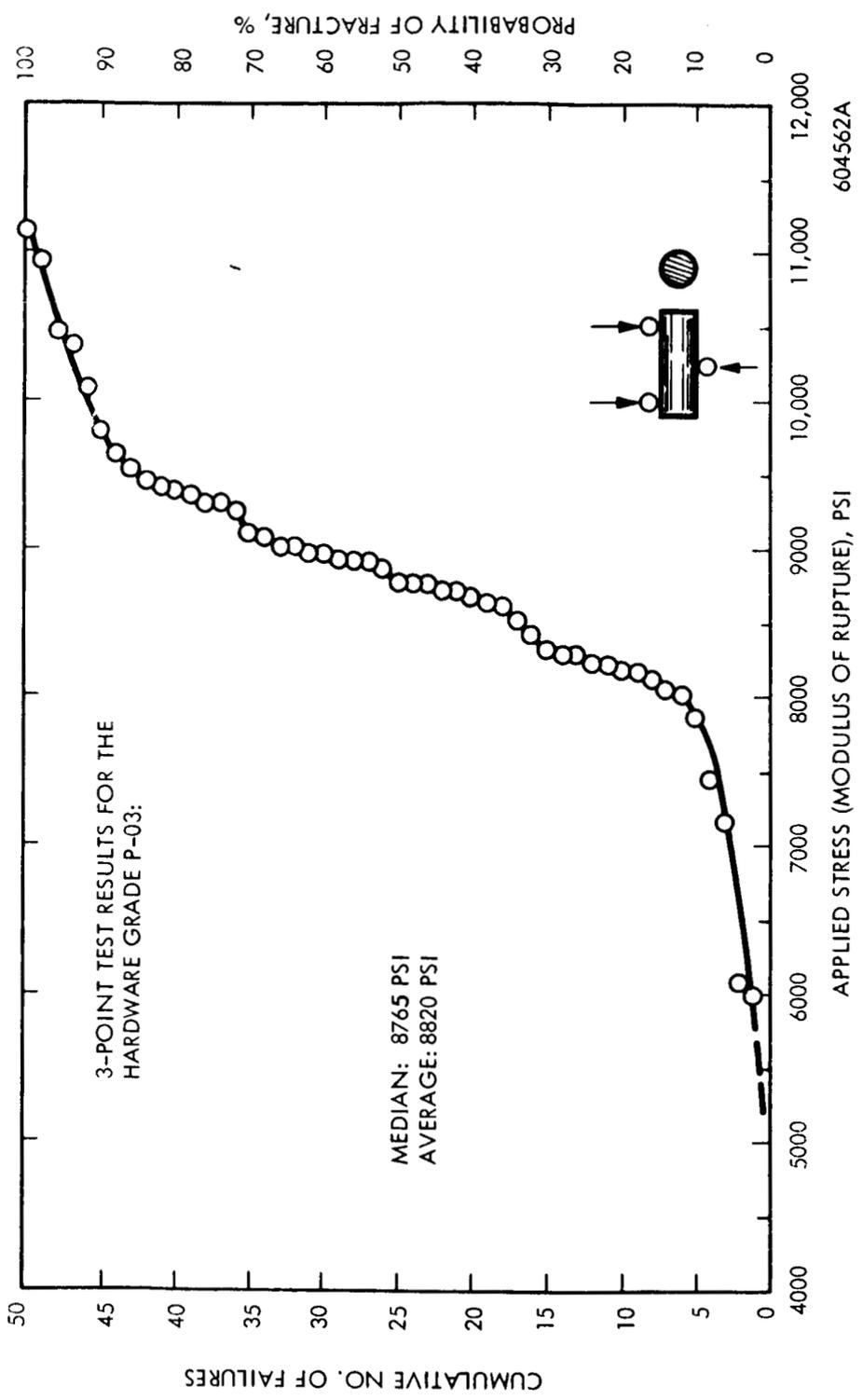


FIGURE 18 STRESS VERSUS FAILURE PLOT FOR THE THREE-POINT FLEXURE TEST ON THE ROUND WITH-GRAIN SPECIMENS OF P-03 GRAPHITE.

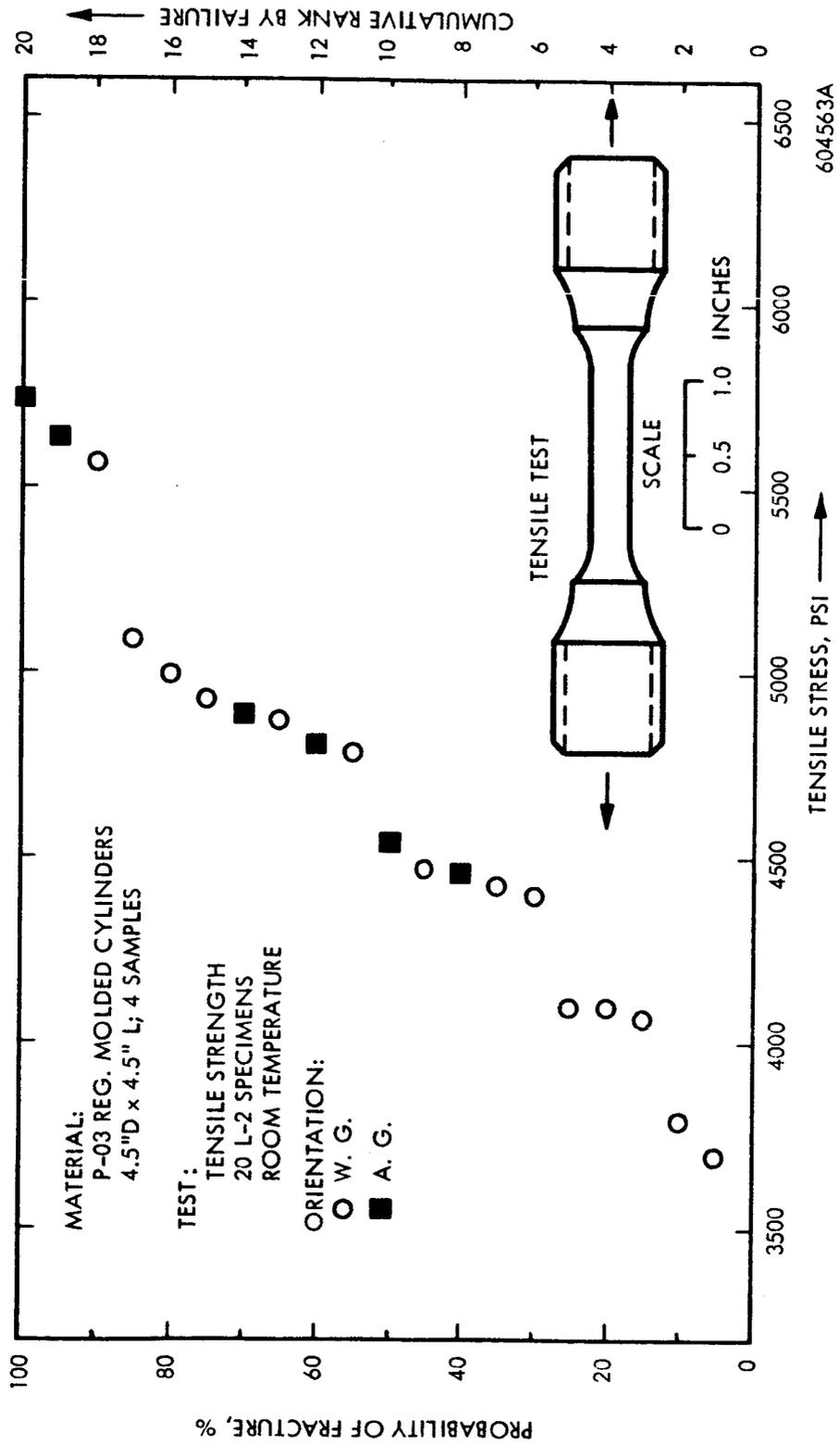


FIGURE 19 STRESS VERSUS FAILURE PLOT FOR THE TENSILE TESTS ON REGULARLY MOLDED CYLINDRICAL STOCK OF P-03 GRAPHITE.

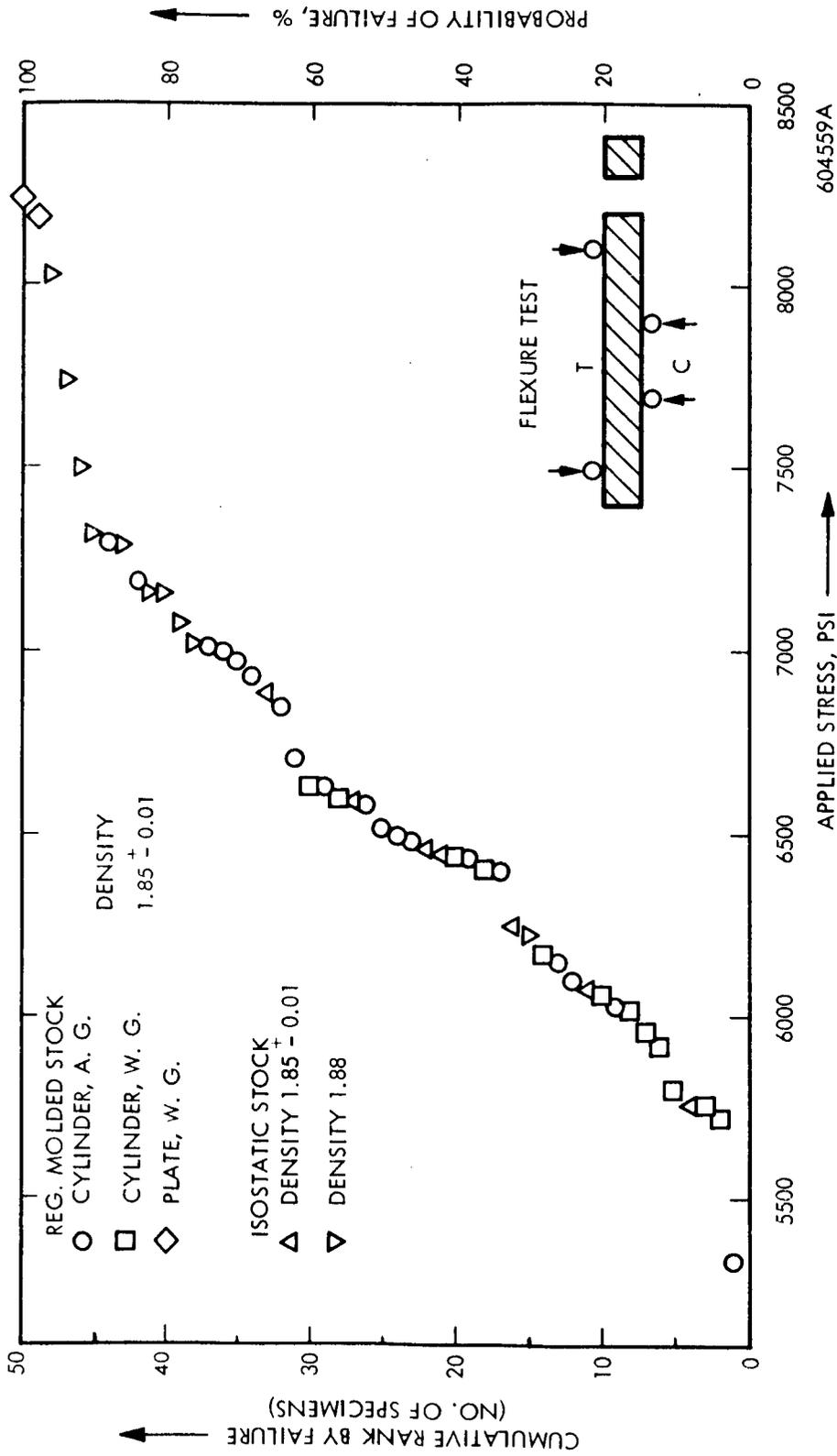


FIGURE 20 STRESS VERSUS FAILURE PLOT FOR THE FLEXURAL TESTS MADE ON THE VARIOUS P-03 SPECIMENS WITH A SQUARE CROSS-SECTION. ALL TESTS WERE CONDUCTED IN FOUR-POINT LOADING.

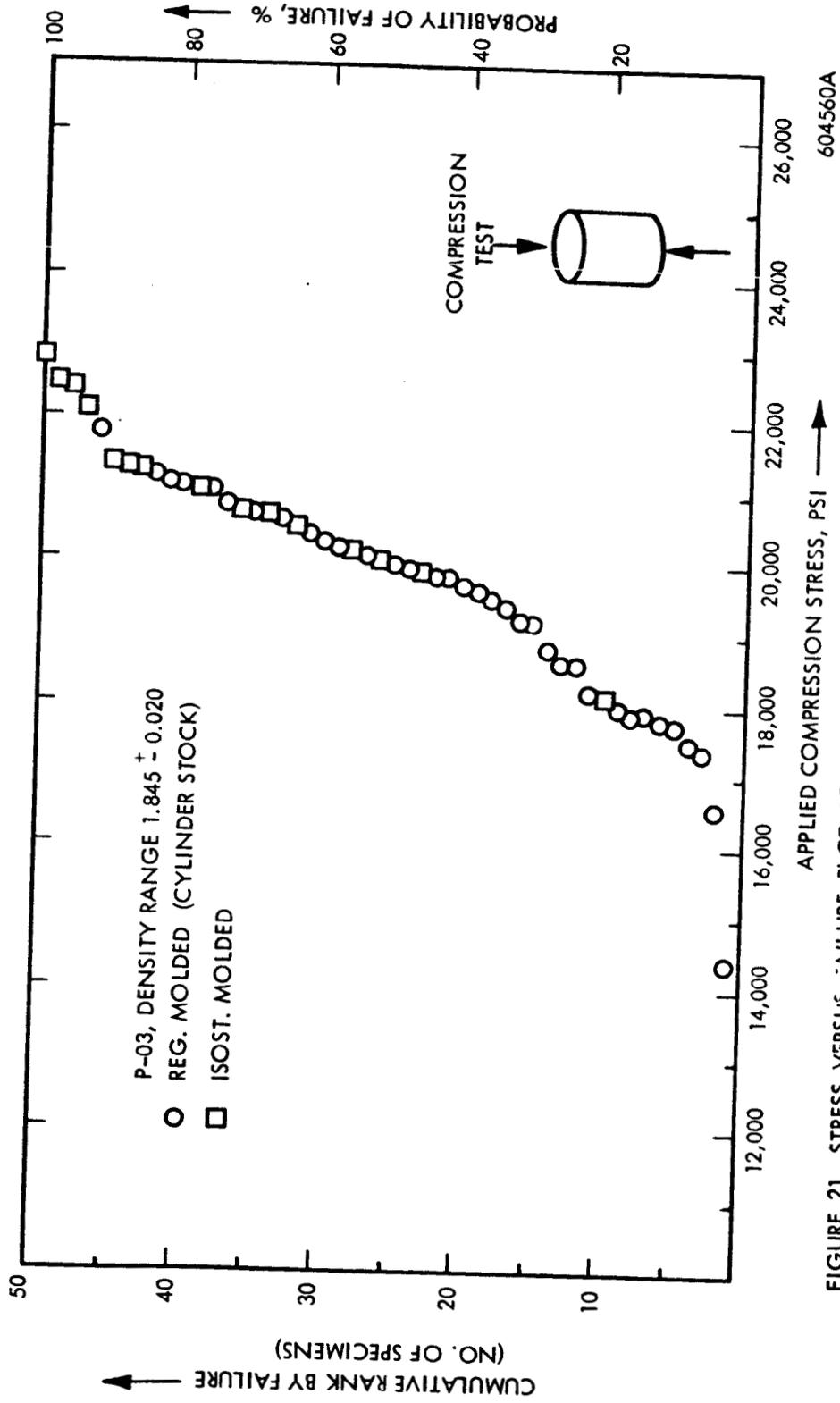


FIGURE 21 STRESS VERSUS FAILURE PLOT FOR P-03 GRAPHITE WHEN TESTED IN COMPRESSION.

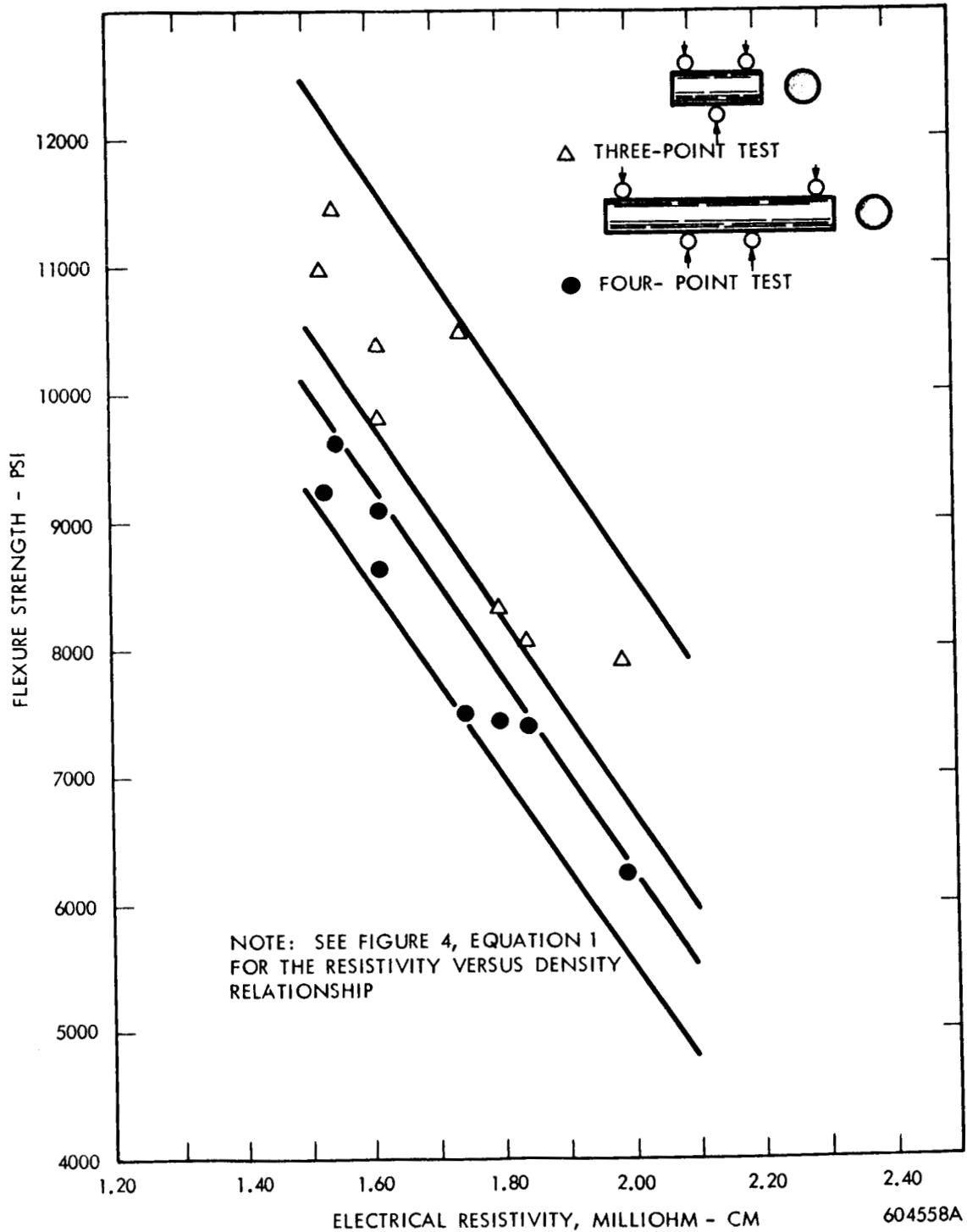


FIGURE 22 CORRELATION OF FLEXURE STRENGTH WITH ELECTRICAL RESISTIVITY FOR ROUND SPECIMENS. THE SPREAD IN RESULTS IS DUE MOSTLY TO SURFACE IMPERFECTIONS WHICH AFFECT THE STRENGTH BUT NOT THE RESISTIVITY.

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2. V. Kachur, Properties of Fiber-Base Graphites, WANL-TME-831, June, 1964
3. V. Kachur, Structural Graphites with High Thermal Expansion, WANL-TME-891, Sept., 1964
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5. The manufacturer is Pure Carbon Company, Incorporated, St. Marys, Pennsylvania
6. V. Kachur, Properties of ATJ Graphite, WANL-TME-956, November, 1964
7. These tests were performed by the Industrial Laboratory Services Corporation under the direction of Mr. W. D. Zeik
8. C. D. Pears, "A Progress Report on a 6500<sup>o</sup>F Furnace for Thermophysical Property Studies", Proceedings, Third Conference on Thermal Conductivity, Gatlinburg, Tenn; Volume 1, pp. 453-479, 1963
9. R. B. Dull, Physical Properties of Some Newly Developed Graphite Grades, WADD-TR-61-72, Volume 26, May, 1964
10. Personal communication from R. R. Paxton, Chief Engineer, Pure Carbon Company
11. W. B. Shook, Critical Survey of Mechanical Property Test Methods for Brittle Materials, ASD-TDR-63-491, July, 1963